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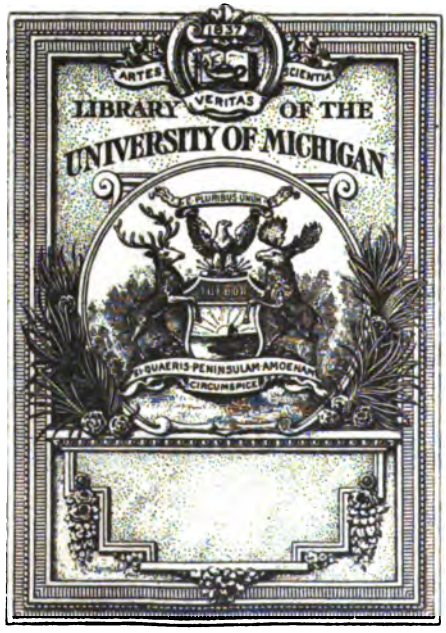
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SOAP AND CANDLES

LUBRICANTS AND GLYCERIN

A TREATISE ON

THE MANUFACTURE

OF

SOAP AND CANDLES

LUBRICANTS AND GLYCERIN

BY

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PREFACE

TO

THE SECOND EDITION.

In consequence of the continued demand for this book, which has been out of print for several years, the publishers have felt called upon to produce a second edition. They deeply regret that the author (who died 23rd December, 1890) should not have been spared to see the numerous signs of the appreciation with which his work has been received.

Being deprived of his assistance and advice, they believe that they are acting in accordance with the course he would have pursued, and in the interest of readers, in availing themselves of this opportunity of revisal to alter or amplify the original text to the extent necessary to render this edition a faithful record of our present knowledge and practice in connection with the various subjects of this volume.

They therefore entrusted the general revisal of the work to Mr. Henry Leask, author of the article on Paraffin in 'Spons' Encyclopædia,' whose great practical experience in paraffin refining and in candle making is sufficient

guarantee for the accuracy of the sections of this work devoted to these subjects.

Our thanks are also due to Mr. J. VEITCH WILSON, who has written, especially for this edition, the chapter on Lubricants.

E. & F. N. SPON.

PREFACE

TO

THE FIRST EDITION.

THE substance of many of the following pages has appeared in various articles under their respective headings in Spons' 'Encyclopædia of Manufactures and Raw Materials,' published in 1882—articles to which the present writer largely contributed, and for one of which ("Soap, Railway-grease and Glycerin") he is solely responsible.

In the present work, however, the whole subject-matter has been re-arranged and very carefully revised. Great pains have been taken to incorporate the latest trustworthy results obtained in the various branches of which the text treats, and some new illustrations have been added.

The aim of the writer, who was practically engaged in these industries for several years, has been, throughout, to make the work as complete and reliable a guide as possible to them, and to indicate briefly but clearly the scientific basis upon which alone they can be successfully conducted. In the chapter upon Theoretical Principles, a certain acquaintance with the elements of chemistry and with the language of symbols is assumed, since what is there stated is intended to serve the purpose of a useful summary,

rather than to be a complete exposition of the theory of saponification.

The author desires to express his thanks to several friends for assistance rendered to him during the production of the book, and especially to Mr. A. H. Allen, to Mr. Calderwood, to his brother Mr. R. Forbes Carpenter, to Mr. C. F. Cross, Mr. Leopold Field, Dr. Perkin, Mr. P. J. Worsley, and Dr. C. R. A. Wright for their kindness in revising the proofs of certain chapters.

WM. LANT CARPENTER.

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Table of the Equivalents and Atomicities of the more common Elements, with Symbols.

Element.	Symbol and Atomicity.	Atomic Weight.	Element.	Symbol and Atomicity.	Atomic Weight.
Aluminium . .	Al^{iv}	27·5	Lead . . .	Pb^{iv}	207
Barium . . .	Ba^{ii}	137	Magnesium .	Mg^{ii}	24
Bismuth . . .	Bi^{v}	208	Manganese. .	Mn^{vi}	55
Bromine . . .	Br^{i}	80	Mercury . .	Hg^{ii}	200
Calcium . . .	Ca^{ii}	40	Nitrogen . .	N^{v}	14
Carbon . . .	C^{iv}	12	Oxygen. . .	O^{ii}	16
Chlorine . . .	Cl^{i}	35·5	Phosphorus .	P^{v}	31
Chromium . .	Cr^{vi}	52·5	Potassium . .	K^{i}	39·1
Copper . . .	Cu^{ii}	63·4	Silicon . . .	Si^{iv}	28
Fluorine. . .	F^{i}	19	Sodium. . .	Na^{i}	23
Hydrogen . .	H^{i}	1	Sulphur . .	S^{vi}	32
Iodine . . .	I^{iii}	127	Tin	Sn^{iv}	118
Iron	Fe^{vi}	56	Zinc . . .	Zn^{ii}	65·2

United States fluid gallon = ·832544 English gallon.
English Imperial „ = 1·201138 United States gallon.

To reduce grammes to grains,		multiply by	15·432.
„	grains to grammes	„	·0648.
„	ounces to grammes	„	28·349.
„	kilogrammes to lbs.	„	2·2046.
„	litres to gallons	„	·22.
„	gallons to litres	„	4·548.

Thermometers.

To convert Centigrade degrees into Fahrenheit degrees, multiply by 9, divide the product by 5, and add 32.

To convert Fahrenheit into Centigrade, subtract 32, multiply by 5, and divide by 9.

Corrigenda.

Page 81. Foot-note, *for* 80–81 *read* 84–5.

„ 110. 17th line, *for* three or four *read* 13 or 14.

„ 396. 2nd line, *for* gr. *read* grm.

SOAP AND CANDLES,

LUBRICANTS AND GLYCERIN.

CHAPTER I.

HISTORICAL EPITOME AND REFERENCES.

FROM the remotest times of which we have any record, the art of softening water, and of using some substance with it, for the purposes of washing and cleansing, appears to have been known and adopted. The elder Pliny, who died A.D. 79, gives us the earliest account of soap, as having been first manufactured by the Gauls, the caustic alkali being produced from wood ashes and natural earths, presumably lime. He also states that it was usually made from tallow and ashes, the best being made from goats' suet and beechwood ashes; he was further acquainted with a lead soap or plaster, and its use in the healing art.* The remains of a soap-factory, with soap in a state of perfect preservation, are still shown in Pompeii. The process of making an alkaline lye was mentioned by Aristophanes (434 B.C.), and by Plato (348 B.C.). Moreover in the Sacred Scriptures, in Jeremiah ii. 22, we read, "For though thou wash thee with nitre and take thee much sope." The Hebrew word *borith* used in this passage is

* The quotations from Pliny are thus given by C. F. Cross in his Lecture on Soap, at the Health Exhibition in London in 1884:—"Fit ex sebo et cinere. Optimus fagino et caprino; duobus modis, spissus ac liquidus."—"Molybdaena cocta cum oleo, jecinoris colorem trahit. . . . Compositio ejus est libris tribus, et cerae libra una, olei tribus heminis."

stated by authorities to refer to vegetable, i.e. potash, lye, and in the Revised Version the word "nitre" is replaced by "lye," which is obviously the substance meant. In Malachi iii. 2, reference is made to "fullers' sope," and the Hebrew word *nether* is there believed to apply to a mineral lye, i.e. to soda in some form. Soap is mentioned by Geber in the second century, and at a later period is frequently referred to by Arab writers as being not only used for detergent purposes, but also for personal application. No advance was made in its manufacture until the early part of the present century, when the researches of Chevreul into the constitution of fatty bodies, and the manufacture of soda from common salt by Leblanc, established the scientific and practical basis upon which the soap industry is now conducted; these two men may therefore be regarded as its founders.

CHAPTER II.

THEORETICAL PRINCIPLES.

ALTHOUGH soap has been made and used for so many centuries the principles which should guide its manufacture have, as already indicated, only been discovered in quite recent times. A proper comprehension of these principles is indispensable to every one who would become a successful manufacturer, because soapmaking is essentially a chemical operation; but as they can only be dealt with very briefly here, the reader unacquainted with them is recommended to consult any good modern work on chemistry, or, still better, and most certainly if he intends to become a practical soapmaker, he is advised to study the science practically, and to attend a course of illustrated lectures on the industry.

Although, in ordinary parlance, the term "soap" denotes simply that combination of fatty matter with alkali which, by its detergent properties, aids in the removal of grease and dirt in washing (in which sense alone will it be used in this work), it is highly important to remember that "soaps" as a class are, strictly speaking, "salts," using that term in the chemical sense. Every salt is formed from an acid and a base, which have opposite properties, producing by their union a third substance differing from either. Thus nitre, nitrate of potash, or potassium nitrate (all are synonymous) may, in the presence of water,* be regarded as a compound of nitric acid and potash, or—

Salt = *Acid* + *Base*,
Nitre = Nitric acid + Potash,

* These words are added, because nitre, as such, cannot, strictly speaking, be said to contain (and therefore to be composed of) nitric acid

because if nitric acid and potash be mixed, nitre and water are formed, and they may be separated from each other by heat, or by allowing the nitre to crystallise.

All the neutral fats of commerce which are used in soap-making, such as tallow, palm oil, coconut oil, cotton-seed oil, and many kinds of grease, are also, from a chemical point of view, "salts," of which the "base" is (not potash but) glycerin, and the "acid" (not nitric but) a mixture of various "fatty acids," which by proper means may, if desired, be separated from each other, and prepared in a state of greater or less purity. Hence (as before, in presence of water)—

Salt	=	*Acid*	+	*Base.*
Neutral fat (e.g. tallow)	=	Various fatty acids	+	Glycerin.

Theoretically, soapmaking is nothing more than turning out the base glycerin from neutral fats by a strong mineral base, or alkali, such as potash or soda, the whole operation being performed in presence of a considerable quantity of water. Hence—

$$\text{Neutral fat} + \begin{matrix}\text{Solution of}\\ \text{Alkali}\end{matrix} = \left\{\begin{matrix}\text{Fatty acids + Alkali,}\\ \text{i.e. Soap}\end{matrix}\right\} + \text{Glycerin.}$$

As, however, certain oils much used by the soapmaker are already fatty acids, and contain no glycerin (e.g. oleic acid, sometimes erroneously called olein, a bye-product of the candle factory, to be hereafter described), or, strictly speaking, do not contain that group of elements which, associated with water, produce glycerin, the formation of soap from them is simply a direct combination of fatty acids with the proper proportion of alkali, as in the case of the formation of nitre above described. This process will be dealt with in describing special soaps.

While, therefore, chemically speaking, any combination of fatty acids with a mineral base may be regarded as a

and potash. When, however, the elements of water are present, these two bodies may be obtained from it, or nitre + water = nitric acid + potash, $KNO_3 + H_2O = HNO_3 + KHO$.

"soap," in practice no commercial soaps are made except with potash or soda, as only those soaps are soluble in water; all others, such as those formed by the union of fatty acids with lime, baryta, and magnesia, or even with the oxides of the metals, as lead, copper, zinc, &c., are insoluble in water, though some of them are used in pharmacy: a "plaster," for instance, is usually a soap from the fatty acids of soft oils, with lead oxide as a base, and, chemically speaking, is an oleate of lead.

A further acquaintance with the theory of salts will make it clear that, in the case of mineral acids and bases there is a certain definite proportion peculiar to each, in which (or in simple multiples of which) they combine with each other. Thus, to take the lowest proportional whole-numbers, 31 parts by weight of pure sodium oxide require 49 parts by weight of pure sulphuric acid to form the neutral salt sodium sulphate; and, in the operation, 9 parts by weight of water are formed, the hydrogen of which is derived from the acid, and the oxygen from the base.* The combined weight of the products is of course exactly equal to the sum of the weights of the constituents, or—

31 parts soda + 49 parts sulphuric acid = 71 parts sodium sulphate + 9 parts water.

This number, called the "combining proportion" or "equivalent" of each substance, is determined by chemical research. Its relation to the atomic weight of the substance cannot be discussed here, but it can scarcely be too strongly insisted on that the fatty acids have their equivalents also; thus, the determination of the quantity of soda necessary for

* In actual practice, it would be very difficult to use the above weights of pure sodium oxide and pure sulphuric acid, since each of them would absorb water from the air while being weighed out. Instead of these pure substances, commercial or laboratory products containing known percentages thereof would be employed, as, for example, caustic soda and oil of vitriol, and the necessary proportion of each would have to be calculated on the above basis from the known composition of the substance used. The extra quantity of water in the caustic soda and in the oil of vitriol would not affect the result.

their saponification is a matter of calculation, and hence the varying equivalents of the different fatty acids is one part of the real explanation of the fact, well known to manufacturers, that the "yield" of soap is so different from various fats.

To revert for a moment to the combination of sulphuric acid with soda,—considerable heat is evolved in the process, which has its parallel in saponification; further, if either constituent had been in excess, there would have resulted a mixture of neutral sodium sulphate with the remainder of whichever constituent was in excess. The fatty acids are remarkable in this respect, that their combining proportions or equivalents are much higher than those of mineral acids. Thus, to combine with 31 parts of pure soda, 284 parts of stearic acid are required, 282 parts of oleic acid, 256 parts of palmitic acid, and only 200 parts of lauric acid, one of the chief fatty acids of coconut oil. Hence, while tallow gives the least "yield" of anhydrous or dry soap among the fats usually employed, coconut oil gives the most, and palm oil occupies an intermediate position.

What has been said above with regard to 31 parts of soda, applies with equal force to potash, replacing 31 by 47, which is the lowest proportional number for potash.

From the explanation above given, it is easy to calculate the quantity of soda or potash necessary to completely saponify—

100 lb. of	Pure Soda.	Pure Potash.
Tallow	10·50 lb.	15·92 lb.
Palm oil	11·00 „	16·67 „
Coconut oil	12·44 „	18·86 „
Oleic acid	10·52 „	15·95 „

It is obvious that the proportion of alkaline lye* to be used must be regulated by its strength; thus, if lye containing 20 per cent., or $\frac{1}{5}$ its weight, of pure soda be used, 5 times the weights above given must be used for 100 lb. fatty material.

* This word is variously written and pronounced ley, lye, lie, leys, lyes, lee, lees. The form *lye* would seem to be the most common and correct.

It will be convenient here to explain briefly the causes of the differences in the combining proportions of the various fats. Their fatty acids are all composed of the three elements, carbon, hydrogen, and oxygen, combined in different proportions of the two first-named, but nearly all containing the same quantity of oxygen. Most of them may be arranged in a series, known as the "acetic," or sometimes as the "fatty" series, of which the lowest term, formic acid, contains 12 parts by weight of carbon, 2 parts hydrogen, and 32 parts oxygen. The other terms of the series differ from each other by 12 parts carbon and 2 parts hydrogen. The table on p. 8 gives some of the principal terms of this series, $C_nH_{2n}O_2$, from which it is clear that the differences in their equivalents are due to the differences in the quantities of carbon and hydrogen entering into their composition, which also affect their melting- and boiling-points.

The fatty acid of the fluid constituent of most natural fats and oils, called oleic acid, belongs to another series, $C_nH_{2n-2}O_2$, known as the "acrylic," of which only a few terms are known; its formula is $C_{18}H_{34}O_2$, and its equivalent is 282. Another term of this series is brassic acid, $C_{22}H_{42}O_2$, one of the chief constituents of rape oil.

A slight acquaintance with the theory of salts in mineral chemistry shows that there are certain acids, such as phosphoric acid, which require three equivalents of base to every one of acid to completely saturate them, and to form a neutral salt. Such acids are called Tribasic. In like manner, it will shortly be shown that *three* equivalents of a fatty acid are required to form a neutral fat with *one* equivalent of glycerin, which may therefore be called a "Tri-acid base." This being so, the process of the saponification of a neutral fat by caustic soda may be represented thus:—

(3 equivalents, stearic acid and soda.)

$$C_3H_5(C_{18}H_{35}O_2)_3 + 3NaHO = 3(C_{18}H_{35}O_2Na) + C_3H_5(OH)_3$$

Tristearin.		Caustic soda.		Sodium stearate or soap.		Glycerin.
890	+	120	=	918	+	92

(1 equivalent, glycerin.)

TABLE A.—THE CHIEF FATTY ACIDS OF THE SERIES $C_nH_{2n}O_2$ OCCURRING IN NATURAL FATS.

(Some rare intervening terms are omitted.)

Name.	Formula.	Equivalent.	Fusing point.	Boiling point.	Natural Fat.
Formic acid	CH_2O_2	46	21° F. (−6° C.)	221½° F. (105° C.)	
Acetic „	$C_2H_4O_2$	60	62½ F. (17 C.)	242½ F. (117 C.)	Cod-liver oil.
Butyric „	$C_4H_8O_2$	88		327 F. (164 C.)	Butter.
Caproic „	$C_6H_{12}O_2$	116	16 F. (−9 C.)	392 F. (200 C.)	Volatile acids of coconut oil; butter.
Oenanthylic acid	$C_7H_{14}O_2$	130		413½ F. (212 C.)	
Caprylic „	$C_8H_{16}O_2$	144	59 F. (15 C.)	457 F. (236 C.)	
Pelargonic „	$C_9H_{18}O_2$	158		500 F. (260 C.)	
Capric or Rutic acid ..	$C_{10}H_{20}O_2$	172	86 F. (30 C.)	..	
Cocinic acid	$C_{11}H_{22}O_2$	186	95 F (35 C.)	..	
Lauric „	$C_{12}H_{24}O_2$	200	109½ F. (43 C.)	..	Fixed acids of coconut oil; spermaceti.
.. ..	$C_{13}H_{26}O_2$	214	116½ F. (47 C.)	..	
Myristic acid	$C_{14}H_{28}O_2$	228	129 F. (54 C.)	..	
.. ..	$C_{15}H_{30}O_2$	242	133 F. (56 C.)	..	
Palmitic acid	$C_{16}H_{32}O_2$	256	145 F. (62 C.)	..	Palm oil.
Stearic „	$C_{18}H_{36}O_2$	284	156½ F. (69 C.)	..	Tallow.
Cerotic „	$C_{27}H_{54}O_2$	410	174 F. (79 C.)	..	Bees'-wax.
Melissic „	$C_{30}H_{60}O_2$	452	192 F. (89 C.)	..	Carnauba wax.

It will be noticed that in this decomposition, the elements of water are taken up, so as to form glycerin. So, also, where a soap is decomposed by any mineral acid, the hydrogen replaces the alkaline metal (usually sodium), and water again takes part in the reaction. Hence (as seen in the table on p. 11), the sum of the fatty acids and glycerin obtained from a neutral fat considerably exceeds the original weight of the fat. The term "hydrolysis" is now often applied to such decompositions of neutral oils.

It has been said that neutral fats may be regarded chemically as "salts," in which glycerin is the base, and a series of complex fatty acids are the acid, and that the process called saponification consists in displacing the base glycerin by the stronger base soda or potash. There is, however, another point of view from which neutral fats may be considered, viz. as the compound ethers of the trivalent alcohol, glycerin. This point will be best illustrated by a reference to the relations between ordinary alcohol and its compound ethers. For example, when alcohol is saturated with hydrochloric acid gas, and the product is distilled, a colourless ethereous liquid is condensed, known as hydrochloric ether, chloride of ethyl, or ethyl chloride. The reaction may be thus expressed:—

Alcohol	+	Hydrochloric acid	=	Hydrochloric ether	+	Water
$C_2H_5(HO)$	+	HCl	=	C_2H_5Cl	+	H_2O,

from which it will be seen that the difference between alcohol and hydrochloric ether consists in the replacement of the group (HO) by the element chlorine, Cl. It will be observed that in this alcohol there is only one (HO) which can thus be replaced, and hence only one such compound ether can be formed from ordinary alcohol and hydrochloric acid.

If, however, glycerin be similarly treated with hydrochloric acid gas, it will be found possible to replace three successive groups of (HO) with three successive portions of chlorine, and to form three different compounds, whose boiling

points are some distance apart, as is shown in the following table :—

Glycerin.	Water.		Boiling point.
$C_3H_5(OH)_3 + HCl =$	$H_2O + C_3H_5(OH)_2Cl$	or Chlorhydrin	440½°F. (227°C.)
$C_3H_5(OH)_3 + 2HCl =$	$2H_2O + C_3H_5(OH)Cl_2$	or Dichlorhydrin	352½°F. (178°C.)
$C_3H_5(OH)_3 + 3HCl =$	$3H_2O + C_3H_5Cl_3$	or Trichlorhydrin	311° F. (155°C.)

Glycerin, therefore, as a trivalent alcohol, forms three distinct classes of ethers, just as the tribasic phosphoric acid forms three distinct classes of salts, with three different proportions of the same base, or even with three different bases.

Similarly, if acetic acid, one of the lowest terms of the series of acids to which it gives its name (p. 8), be made to react on glycerin, we have the three compounds—

$$C_3H_5(OH)_3 + C_2H_4O_2 = H_2O + C_3H_5(OH)_2 . C_2H_2O_2 \text{ Monacetin.}$$
$$C_3H_5(OH)_3 + 2C_2H_4O_2 = 2H_2O + C_3H_5(OH), 2C_2H_2O_2 \text{ Diacetin.}$$
$$C_3H_5(OH)_3 + 3C_2H_4O_2 = 3H_2O + C_3H_5, 3C_2H_2O_2 \text{ Triacetin.}$$

Now it has been shown by Berthelot, that if the higher terms of the fatty acid series be allowed to react upon glycerin under suitable conditions, a precisely similar series of compounds may be formed in exactly the same way. Hence, if glycerin be regarded as an alcohol containing the trivalent radicle C_3H_5, and stearic acid be taken as an example, the relationship between glycerin and mono-, di-, and tri-stearin may be thus expressed :—

Glycerin.	Monostearin.	Distearin.	Tristearin.
$\left.\begin{matrix} C_3H_5 \\ H_3 \end{matrix}\right\} O_3$	$\left.\begin{matrix} C_3H_5 \\ C_{18}H_{35}O_2 \\ H_2 \end{matrix}\right\} O_3$	$\left.\begin{matrix} C_3H_5 \\ 2C_{18}H_{35}O_2 \\ H \end{matrix}\right\} O_3$	$\left.\begin{matrix} C_3H_5 \\ 3C_{18}H_{35}O_2 \end{matrix}\right\} O_3.$

Although the first two terms of this series, whatever be the fatty acid employed, are merely chemical curiosities, interesting from their constitution alone, the third term in each case is very important commercially, inasmuch as nearly all neutral fats are built up on the lines indicated in that term, or, in other words, tallow is mainly composed of tristearin and triolein, and so on for other fats and oils.

The following short table gives the formulæ, on this view, of some of the more important and illustrative members of the series :—

			Molecular weight.	Products of Saponification of 100 parts. Fatty acid.	Glycerin.
Tristearin	$C_3H_5(OC_{18}H_{35}O)_3$	Tallow	890	95·73	10·24
Triolein	$C_3H_5(OC_{18}H_{33}O)_3$	,,	884	95·70	10·40
Tripalmitin	$C_3H_5(OC_{16}H_{31}O)_3$	Palm oil	806	95·29	11·41
Triricinolein	$C_3H_5(OC_{18}H_{33}O_2)_3$	Castor oil	932		
Trilinolein	$C_3H_5(OC_{18}H_{27}O)_3$	Linseed oil	794		
Tributyrin	$C_3H_5(OC_4H_7O)_3$	Butter	302		
Triacetin	$C_3H_5(OC_2H_3O)_3$	Cod-liver oil	218		

It must be borne in mind, therefore, that the object of the soapmaker is to replace the whole of the glycerin in the tristearin, triolein, &c., of natural fats, by the proper combining proportion or equivalent of either soda or potash, according to the kind of soap desired, and that the more perfectly and the more accurately this can be done, the purer and the more satisfactory will be the ultimate commercial product.

It may be convenient here to point out that for the production of a large class of soaps, and in particular of that known as Yellow, or Household soap, rosin (colophony) is largely employed. Chemically speaking, rosin is a mixture of acids (pinic, sylvic, colophonic, and pimaric) whose general formula is $C_{20}H_{30}O_2$, and their combining proportion (with 31 parts pure soda) is 302. According to another authority, ordinary commercial rosin is said to consist chiefly of abietic anhydride, $C_{44}H_{62}O_4$, from which sylvic acid may be obtained by acting upon it with sulphuric acid. The difficulty, however, of isolating and analysing these complex acids is very great, and the whole subject will well repay a careful investigator. Rosin decomposes alkaline carbonates, and combines instantly with caustic alkalies, forming in each case a so-called rosin soap, which, when soda is employed, is a thick, slimy, brown mass, containing 15·8 per cent. of pure soda; its attraction for water is so

great as to cause it to become semi-liquid on exposure to air, even though previously dried artificially.

It is well known that when many substances, whether elements or compounds, pass from the liquid to the solid state, they assume a more or less crystalline form, characteristic of each substance. Thus, metals crystallise from a state of fusion, and most salts from a state of solution. The same is true, to a greater extent than is generally supposed, in the case of the alkaline salts of the acetic series of acids, i.e. both in hard and soft soaps. The appearances known as "grain" or "strike" in a hard soap, and "fig" in a soft soap, are due to the crystalline character of soap, and are largely influenced by the quantity of water present. Ordinary salts, when crystallising, take up a certain quantity of water, which is definite, invariable, and necessary to the crystalline structure of the salt. When they lose this water of crystallisation in either dry or warm air, they lose also their crystalline structure, and usually become amorphous. The same thing happens, to a less extent, with soap; but, owing to the peculiar state of aggregation of the soap-crystals, the loss of water does not *appear* to be attended by any change in structure.

This difference in their behaviour to water, between the potash and soda soaps of the same fatty acids, is very remarkable, and lies at the root of the distinction between hard (or soda) and soft (or potash) soaps. The soda salt, on exposure to air, will gradually lose nearly all its water, at a rate depending upon the dryness and temperature of the air. The potash salt, on the other hand, never becomes thus dry, but retains enough water to form a soft slimy jelly, and if it is artificially desiccated, it absorbs large quantities of water from the air, often amounting to half as much again as the weight of the dry soap. This difference is best seen in the case of the salts of pure fatty acids, thus :—

100 parts	dry	potassium oleate	absorb	162	parts	water from the air.
100 „	„	potassium palmitate	„	35	„	„
100 „	„	sodium stearate	„	7½	„	„

It will be convenient here to consider another physico-chemical property of soaps, of great practical importance to the soapboiler. The property referred to is the behaviour of soap to various saline solutions, and although not instrumental in saponification, it is almost essential in separating the foreign matter which would otherwise render hard soap impure; it is also influential in controlling the amount of water therein. Although soda soaps are soluble in water, they are not soluble in a solution of common salt, nor of caustic soda. If, therefore, common salt be added to a solution of soap (or even of partially saponified fat) in water, the salt dissolves, and turns the soap out from its state of solution in the form of small flakes, which collect together and float on the surface of the salt solution by virtue of their less sp. gr. The same thing happens when a strong solution of caustic soda is added to soap in an aqueous solution, or, more gradually, when a solution of soap in water containing excess of caustic soda, or some amount of sodium chloride, is concentrated by the evaporation of its water, as when a soap-copper is boiled by fire or close steam. The addition of salt (or of strong lye), therefore, to soap containing an excess of water, removes the superfluous water, and, in chemical language, precipitates the soap from it. Soap so precipitated contains 35–40 per cent. of water. When the fatty matter employed contains coconut or palm-kernel oils, more salt (or soda lye) is required for this operation than when those oils are not used.

It may also be noted here that if common salt, i.e. sodium chloride, be added to a potash soap in solution, an interchange of acids and bases takes place, soda soap being formed and potassium chloride left in the solution. This process has been actually used for the fabrication of hard soaps in Germany, where potash salts have at times been very abundant.

The chemical principles which are involved in the preparation of the lye, or solution of caustic soda or potash, for use in soapmaking, will form the subject of a later chapter.

CHAPTER III.

RAW MATERIALS: THEIR SOURCES AND PREPARATION.

THE choice of the fatty matters to be employed in the fabrication of any particular soap must largely depend upon the kind of manufactured product which it is ultimately desired to obtain. The distinction having been drawn between hard and soft soaps, it may be stated broadly that hard soaps are made from the more solid animal and vegetable fats, with soda as a base, while in the manufacture of soft soap, oils that are fluid or semi-fluid at the ordinary air-temperature are usually employed, and especially linseed and other seed-oils and the cheaper varieties of fish-oils. Certain oils make a hard or a soft soap, according as the alkali employed is soda or potash, or, in some cases, a mixture of both. As a general rule, not more than ¼ of the potash in a soft soap can be replaced by soda (for cheapness sake) without impairing the quality of the soap. No mineral oil yet discovered is capable of saponification in the true sense of the word, and hence its presence must be carefully guarded against in the cheaper fats and oils often employed by the soapmaker. Hard soaps invariably contain soda as their base, and, *ceteris paribus*, the hardness varies inversely as the quantity of water in the soap. An oil as fluid as commercial oleic acid will produce a hard soap with soda, if the process be so conducted that but little water is present during the operation. Tallow, mixed with varying proportions of rosin, is employed for household "yellow" soap; kitchen grease, bone fat, discoloured lard and other greases, with rosin, for inferior grades of the same; while the same greases, and palm, cotton-seed, olive, coco-

nut, and palm-kernel oils, are used either alone, or in various combinations, for the different kinds of mottled soap. The raw materials of soaps for special manufacturing, toilet, and other purposes, will be mentioned when treating of those soaps. As the market values of the most important fats, such as tallow, lard, and palm oil, vary considerably, and largely influence the prices of inferior grades of saponifiable materials, the price of any particular fat will naturally determine its employment or otherwise, provided that it is suitable in other respects for the particular purpose in view.

This chapter will contain a short account of the sources and modes of preparation of the chief saponifiable materials as they occur in commerce; the succeeding one will be devoted to an account of the processes usually employed to clarify, bleach, or otherwise prepare these materials for the soap-pan; and that will be followed by one containing instructions for the proximate analysis of these substances, and for the detection of adulterations in them.

SECTION A.—ANIMAL FATS.

Tallow.—Of all fatty matters used in the production of household soaps, by far the most important is Tallow. The cellular tissues of man and quadrupeds contain a concrete fat, the whole mass of tissue and fat being known as "suet." The term "tallow" is applied to this fat when it has been liberated from the tissue. Commercially, tallow is obtained almost solely from the ruminant animals, sheep and neat cattle, and is produced chiefly in the essentially pastoral portions of the globe. In many cases the animals are (or were) reared more for the sake of their tallow than their flesh, and in Australia millions of them have been boiled down as they were killed, the boiled flesh being used for pig-feeding or manure. Recent improvements in transporting meat will doubtless prevent the recurrence of such a wasteful process, though the tallow may retain its importance as a commercial product, and should be prepared at the places

where the animals are killed for despatching to a distance in cold chambers.

The process of separating the fatty matters from the animal tissue which contains them is usually termed "rendering," as applied to the fats themselves. These fatty matters are contained in animal tissue, into whose composition water largely enters. The simplest method of separating them from each other is by heating the fat in an open pan over a fire, at a temperature considerably above the boiling-point of water, 212° F. (100° C.), constantly stirring the mass, whereupon the animal tissue dries and cracks, allowing the pure fat to run out, and become separable by mere straining. This process, however, requires careful watching, as the tallow is apt to be discoloured by overheating, and, unless the fat be very new, it is certain to evolve noxious odours.

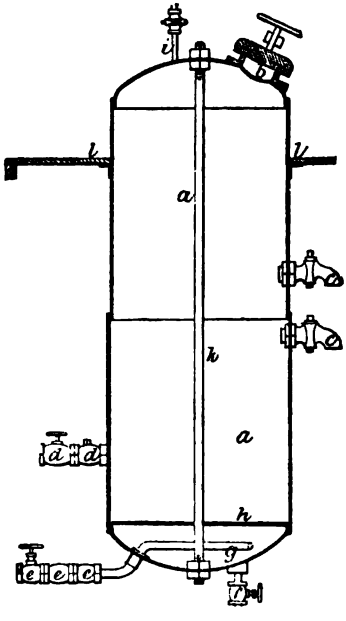


FIG. 1.—RENDERING-BOILER.

The methods employed on a large scale may be divided into two, according as it is desired to save the animal tissue in a solid form, or not. If it be so desired, a very suitable apparatus is that used by Dole, of Bristol, and made by Miles, engineer, Bristol, shown in Fig. 1. The apparatus consists of a strong iron cylinder *a*, provided above with a charging hole *b*, closed by a sliding cover; a man-hole near the bottom for the discharge of solid refuse; two taps *c* for

drawing off pure fat; water feed-valves *d*; steam feed-valves *e*; and water draw-off valve *f*. Steam at a pressure of 60–80 lb. is introduced at *e*, and circulates in the coil *g* below the perforated false bottom *h*, *d* supplying water when necessary. The vent-tap *i* regulates the pressure and permits the blowing off of steam, The apparatus being charged, steam is introduced for 6–8 hours. With a heavy charge, the fat can be drawn off by the upper of the taps *c*, being floated to that level if necessary by letting in water. With a light charge, it is considered better to draw off all liquids together by the lower tap *c*, and skim off the fat when cold. The apparatus is strengthened by a stay-bolt *k*, and has a stage at *l*.

When it is desired to obtain the very finest and purest tallow, for the manufacture of toilet soaps of the first quality, some modification of one of the three following methods, which are used in the early stages of the manufacture of butterine, may be adopted with advantage. The residual matters, however, in all cases contain some amount of inferior tallow, and they should therefore be subsequently "rendered" by one of the ordinary processes.

A method which is very extensively employed in the United States consists in thoroughly washing the picked beef suet in water, and placing it in a steam-jacketed pan; the contents are never allowed to experience a temperature exceeding 120° F. (49° C.). The pure fat that runs off is "seeded" (i. e. allowed to cool very slowly, which facilitates the mechanical separation of the stearin) and pressed. The resulting stearin may be sold for candle-making purposes, while the liquid portion (oleomargarin) is fit for the manufacture of butterine, or for any purpose for which a very pure fat is required.

Another arrangement is adopted by W. Cook and S. Hall, of the East London Soap Works, Bow. The freshest beef suet is first thoroughly disintegrated and reduced almost to a pulp. A wooden chamber is provided, of sufficient height to accommodate a workman, and with a passage up the centre. At the two sides are inclined racks; upon these, shallow

iron trays are slid in from the outside, and rest with a slight slope towards the central passage. Along the lower free edge of each row, is an open iron gutter, leading to a receiver outside. The comminuted fat is laid in thin layers upon the trays, and the temperature of the chamber is raised by steam-pipes to such a degree as will just suffice (and no more) to liquefy the fat, which then escapes by the gutters to the receiver, leaving behind such shreds and portions of tissue as are not liquefied by the heat. The temperature should never exceed 130°–135° F. (54°–57° C.), and a much lower degree will be equally effective if sufficient time be allowed.

The third process originated from a surmise of the eminent French chemist Mège-Mouries, that the formation of butter contained in milk was due to the absorption of fat contained in the animal tissues. This led to the experimental splitting-up of animal fats, with the result that a process was devised by which the olein and margarin contained in the fat could be almost completely separated on a commercial scale from the stearin. This process consists in heating finely minced beef suet with water, potassium carbonate, and finely chopped fresh sheep's stomachs, at a temperature of 113° F. (45° C.), when the combined influence of the pepsine of the sheep's stomachs, and the heat, causes the separation of the fat from the cellular tissue. The fatty matters are removed, cooled, and submitted to powerful hydraulic pressure in the cold, which effects their separation into a solid and a liquid portion, the former being stearin, a commercially valuable article, and the latter the olein and margarin, or oleomargarin, required for making the artificial butter. This is the process patented by Mège-Mouries, and under which a large quantity of butterine is made. The disadvantages of all these processes are the necessity for the comminution of the fat, and the length of time required by the process, i. e. the large amount of plant necessary to "render" a few tons of rough fat. Their advantages are the almost complete freedom from noxious vapours (hence they are strongly recommended by the Government factory inspectors), and the great purity of the product.

When the saving of the animal tissue is a matter of no moment, it is better to "render" the fat in the presence of water and steam. This can only be completely done under slight pressure (boiling the comminuted fat upon water in an open vessel only extracts part of the tallow, &c.), and is most suitably effected in a vessel such as that shown in Fig. 2. The apparatus consists of a series of steam-tight cylinders of 1200–1500 gal. capacity, formed of boiler-plate, with a length about 2½ times as great as the diameter, and provided with false bottoms. The operation proceeds thus:—The cylinder is fed through the manhole K with crude fatty matters to within about 2½ ft. of the top. The manhole is secured, and steam is admitted by the foot-valve into the perforated pipe C. The safety-valve O is set at the required pressure, and frequent testing of the state of the contents of the cylinder is made by opening the try-cock R. An excess of condensed steam in the cylinder will be indicated by the spurting ejection of the fatty matters, when the regulating-cock X must be opened, and the condensed steam be drawn off into the tub T, till the escape of fatty matter from R has ceased. After 10–15 hours' continued steaming, the steam is shut off, and such as remains uncondensed in the cylinder is let out by the try-cock and safety-valve. The fatty matters then gradually separate out and form the uppermost layer, and are drawn off through the cocks *p* into ordinary coolers. The fat being emptied from the cylinder, the cover F is raised by the rod G from the discharging hole E, and the residue falls into the tub T. Should the residue retain any fat, it is returned to the cylinder with the next charge. The pressure of steam commonly used is 50–75 lb. a sq. in., sometimes advancing to 100 lb., though this last figure is excessive, and calculated to injure the quality of the fat by decomposing the animal matters present. The yield obtained is considerably greater than by the ordinary methods, being stated at 12 per cent. extra for lard, and 6 per cent. for tallow. On the other hand, it is almost necessary to wash the fat with fresh water, and

remelt and settle or strain it, to remove the last traces of animal matter held in suspension by the unseparated water, and having a tendency to putrefy.

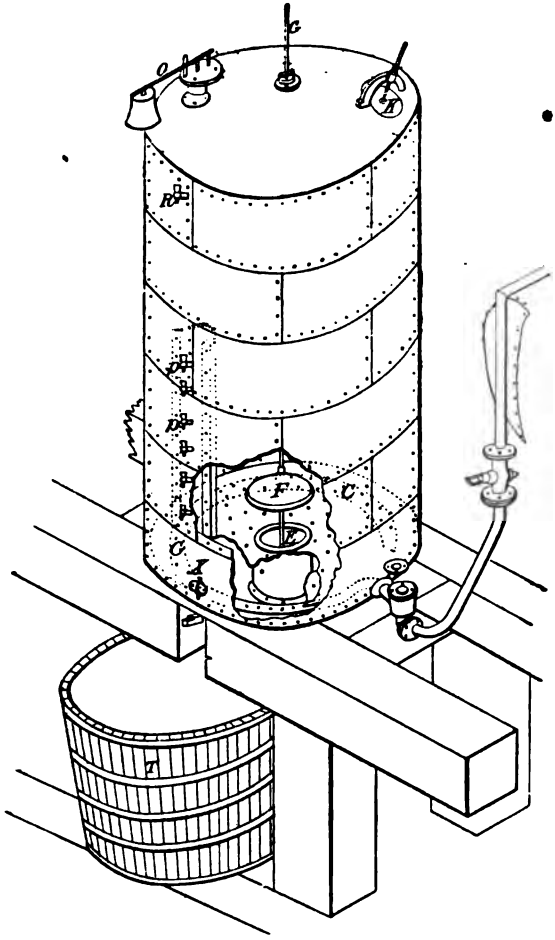


FIG. 2.—RENDERING BOILER.

Another modification of the steam process is to subject the rough fat to the action of about ¼ its bulk of water, containing 2–3 per cent. of sulphuric acid, boiling the whole by steam at atmospheric pressure, i.e. in an open, or loosely closed, lead-lined tank. There exists, however, a prejudice against tallow or lard in whose preparation any "chemicals" have been used.

In all cases it is highly desirable to render the fat as soon as possible after its removal from the animal's body, since the animal tissue rapidly decomposes and sets up fermentation, producing rancidity in what would otherwise be a neutral fat, and injuring the colour, &c. of the ultimate product. The different kinds of fat should also be sorted, and each description melted separately. In large establishments, such as the Union stockyards of Chicago, the stockyards at St. Louis, and the packing-houses of Cincinnati, where thousands of beasts are killed every day, there is a row of these digesters, each one allotted to its own kind of fat, which is placed therein a few minutes after the death of the animal. In this way is secured great uniformity in the various grades of tallow and lard produced. The waste liquor, containing large quantities of nitrogenous matter, is sold for manure.

Many soap-makers "render" rough fat in a soap-copper, by the simple action of wet steam upon it, while the cover of the copper is secured by bolts or weights. When all the tallow has been skimmed off, a weak impure solution of caustic soda and common salt, technically known as "half-spent lye," is run in, and the whole is boiled. The object is to saponify the last remaining portions of tallow, and the discoloured imperfect soap is used in lower grades of the pure article. The process, however, is a wasteful one, since large quantities of the soda are used up in forming ammonia, by its action upon the nitrogenous animal tissue, and this ammonia, being volatile, escapes with the noxious vapours, and thus a valuable fertilising agent is lost. As far as concerns the avoidance of noxious odours, rendering by

steam has undoubted advantages, and it is altogether a more rapid and satisfactory process.

The foregoing descriptions of the various processes for the "rendering" of beef, mutton and pig fats (for the production of lard), apply equally, *mutatis mutandis*, to that of kitchen stuff, ships' grease, &c. In these cases, however, the liability to rancidity, and production of noxious fumes, is much greater than with fresh fat.

The nature and qualities of commercially pure tallow vary greatly. The most important constituents are stearin and olein; stearin predominates, but its proportion fluctuates with the species, age and sex of the animal, the portion of its body which afforded the suet, and the nature of its food. Ordinary fodder produces the hardest tallow, while animals fed on brewery refuse, &c. give quite a soft fat. Beef tallow usually contains less stearin than does either mutton or venison, and mutton tallow is always whiter than beef tallow, but S. American beef tallow presents the curious exception of containing *more* stearin than S. American mutton tallow. The hardness and melting-point have an equal influence upon the value of the tallow, and exhibit the same want of constancy under similar changes of condition. The degree of solidity is largely influenced by the food, increasing as the latter is drier. Pure tallow is white and almost tasteless, but much of that imported has a yellow tint. It dissolves in about 40 parts of boiling alcohol, sp. gr. 0·82. Nearly half its weight is olein, the remainder being chiefly stearin; 100 parts of it yield by saponification 94·5 parts of fatty acids, whose solidifying point varies from about 110° to 122° F. (43°–50° C.), according to the locality and the circumstances of its production. According to Dr. Hager, its specific gravity varies thus, at 60° F. (15°·5 C.):—Beef, 0·925–0·929; mutton, 0·937–0·940; beef and mutton mixed in equal proportions, 0·936–0·938. When exposed to the air, it gradually undergoes the change known as rancidity, becoming very white, and developing a peculiar and character-

istic smell. In this condition it should be carefully avoided by the soap-maker, because the acids which are formed produce brownish-red compounds with soda, seriously discolouring the soap, or in technical phraseology, the rancid tallow "works foxy." No satisfactory process has yet been discovered for removing these complex acids from tallow, hence tallow which is contaminated by them should be relegated to the candle-maker, for even as a lubricant it would be very objectionable in use, owing to the action of the free acid upon the metal.* It is worthy of note that the more perfectly a tallow is rendered, the less liable is it to undergo this change, since the minute portions of nitrogenous matter left in it appear to act as ferments in promoting those chemical changes, chiefly caused by oxidation, which result in the formation of these complex acids.

In commerce, tallow occupies a very important place. Russia exports immense quantities, chiefly from the ports of Cronstadt, Odessa and Taganrog. Twenty years ago, Russia's annual production was reckoned at 160,000 tons, half of which was consumed locally. The home consumption has since much increased. Russian tallow is nearly all beef, and comes chiefly from Siberia and the Ukraine. It is transported in casks of 300–400 *kilo.* The commercial quotation of "P.Y.C. tallow" (Petersburg Yellow Candle) is a fiction, and does not regulate the market price of tallow; it is a mere speculative medium, thousands of casks being bought and sold that have no existence whatever. Russian tallow has lost much of its hold on the market, and now forms but a small item in the total consumption in this country, notably in the case of the soap and stearin makers, for whose purposes it is less suited than for "dips." The States of S. America afford very large quantities of tallow from the carcasses of animals slaughtered principally for the sake of this product and their skins, bones and horns. It is generally known as "River Plate" tallow, and is mostly shipped

* This will be further alluded to in the chapter on Lubricants.

from the Rio de la Plata. It has a strong yellow colour, but is of good quality; it first arrived in serons of hide, but now comes in old wine-casks—pipes and half-pipes. The United States ship considerable quantities of tallow to Europe, chiefly from New York and New Orleans. It is usually known in the trade as North American, and as Western, tallow, the former being packed in large drums, and the latter usually in barrels. Western tallow is frequently irregular in colour, but much harder as a rule than the other variety, which is too often mixed with softer fats, and the whole sold as "Prime Butchers' Association Tallow." Another great source of tallow, especially in recent years, is Australia, many hundreds, and even thousands of casks, of colonial tallow being disposed of at the weekly public sales in London. The production of tallow in the United Kingdom has been estimated at 100,000 to 120,000 tons yearly. Small supplies occasionally are received from China and Japan, the East Indies, the tallow from whence is very hard but bad in colour, from Turkey, Holland, Belgium, the Falkland and Sandwich Islands, and many other places. Our total annual imports of tallow and stearin fluctuate between 50,000 and 65,000 tons.

Lard and Lard Oil.—The fat of the pig, freed from the cellular tissue in which it is contained, is known as "lard." The pieces of adipose tissue are sometimes salted a little to keep them sweet, and are stored in barrels. They are scored and sliced till they do not exceed about 1 in. in thickness, and are then thrown into caldrons. The common methods of "rendering" the lard, among very small fat-melters, is by means of boiling with water in an open cast-iron vessel exposed to the direct heat of a fire. The use of a steam-jacketed pan and injected steam, as described on p. 19, is universal in the great American centres. Whatever plan be pursued, the oil is liberated from the tissues, and forms a layer on the surface of the mass; it is drawn off while still warm and liquid, and received in the vessels in which it is to be stored and transported. These vessels are bladders in

the case of superior qualities, and little wooden kegs for inferior sorts. The fat immediately surrounding the kidneys yields the best and purest lard. This, and that which is obtained in flaky layers between the flesh and the skin of the animal, is known as "leaf" lard, and is kept separate from the rest, being much more valuable—harder and less fusible. Second quality lard (which, in reality, is the ordinary commercial first quality of wholesale quotations) is used for the production of lard oil; the third quality, from trimmings which have become slightly tainted, is employed for making low-grade oil or for soap. The best pure lard should be moderately firm and white; the degree of firmness entirely depends upon temperature and the molecular condition [unless stirred while cooling, or exposed to very great cold in a refrigerating room, it is usually "seedy" and sloppy, even at as low a temperature as 50° F. (10° C.)]; when melted, it is as clear and transparent as water: completely free from taste and smell; liquefiable at about 212 degrees F. (100° C.) without ebullition, or affording a particle of deposit; and containing never more than 2 per cent. of either water or salt (good American lard has no salt, and not above 0·5 per cent. of water). Its composition, according to Braconnot, is 38 per cent. of stearin and margarin, and 62 per cent. of olein; 100 parts of it by saponification yield 9 parts of glycerin and 94·65 parts stearic and oleic acids. The solidifying-point of the fatty acids of lard is about 106° F. (41° C.). Lard dissolves in 36 parts boiling alcohol at 0·816 sp. gr. According to Dr. Hager, the sp. gr. of lard is 0·931–0·932 at 60° F. (15°·5 C.) when fresh, and 0·940–0·942 when old.

The lard produced in the United Kingdom is chiefly Irish. Of European countries, Russia, Hungary, and Servia hold the foremost position. Hungarian lard is supplied to the whole Continent; many of the pigs are so lean as to be useless for food, and some establishments in Budapest boil down ½ million yearly for the lard alone. Pig-keeping is the

leading industry of Servia, and large supplies of lard are now derived from that country. At present America is the chief producer. In the United States, the average yield of lard from each pig was 25 lb. in 1862, and 37⅓ lb. in 1874. Our total annual imports of lard vary between 33,000 and 45,000 tons.

"Lard oil" is prepared by placing the lard in woollen bags between wickerwork and the plates of hydraulic presses, where it is left for some 18 hours under a pressure of about 10 cwt. a sq. in. in the cold. The oil or liquid portion (olein) is thus expressed in a pure, colourless and limpid state, in the proportion of 62 per cent. of the weight of lard. It remains liquid even in the presence of great cold. It is largely used for adulterating olive oil in France, and sperm oil in the E. States of America; it is esteemed as a lubricant, and is said to be also used for illuminating. In Cincinnati, there are some 40 manufactories, turning out about 1½ million gal. of this oil annually, The so-called "lard stearin" left in the presses is frequently used as a substitute for tallow in the soap-pan, when the price of it is suitable.

Horse Grease or Mares' Grease.—Quantities of this article are shipped from S. American ports. It has about the same consistency as ordinary commercial American lard, and, except for its inferior colour, it has practically a like value for the purposes of the soap and candle maker.

Bone Grease or Bone Tallow.—Bones invariably contain a certain amount of fat, which must be extracted as a preliminary to any other process needed to fit them for their various uses, and this fat is so largely employed in the manufacture of soap that it is not uncommon to find the two industries of bone-boiling and soap-making united. All kinds of bones should not be boiled indiscriminately together, both because the bones themselves will be devoted to different purposes, and because the qualities of the fat produced will also be various; bullocks' hollow shank bones yield the best fat. A simple method is to boil the bones

with water in large open vessels for 24 hours, skimming off the fat as it floats. The odours evolved during this process, however, are exceedingly offensive, and a very great improvement consists in the application of steam in closed vessels, as adopted by most large firms. The form used by Morris and Griffin, at Wolverhampton, is shown in Fig. 3. It is composed of a 1⅛-in. iron cylinder *a*, 6 ft. long by 3 ft. 6 in. in greatest diameter, furnished with hinged doors *b c* at top and bottom, which are tightly closed during operations by 1¼ in. screw-threaded bolts and nuts *d*, and further provided with 2-in. tap-holes at *e* and *f*. The bones are introduced at the top door *b*, which is then secured; steam at about 286° F. (141° C.) is introduced for 40 minutes; the steam is shut off, and that remaining in the cylinder *a* is let out into a condenser; ½ hour later, the fat is drawn off by the tap *f* at the bottom of the cylinder; the bottom door *c* is then opened and the bones are allowed to fall upon the floor below. The bones are rendered much more brittle than by ordinary boiling, and are much drier and more easily ground, requiring no previous storing. The cylinder shown will steam at a charge 46½ cwt. of bones, yielding 87–88 lb. of fat per ton.

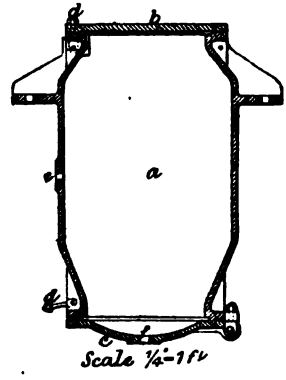
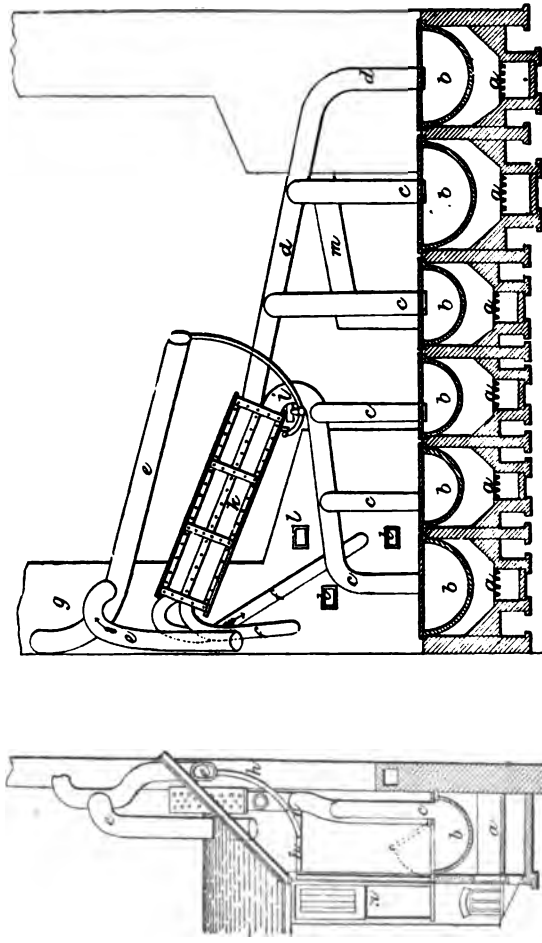


FIG. 3.—BONE BOILER.

One of the best and most inoffensive arrangements of bone boilers for a large establishment is that adopted at Adams' knackery in Birmingham, erected under the direction of Dr. Alfred Hill, and shown in Figs. 4, 5. A set of 6 pans is fixed and heated in the usual way. They are enclosed above in a sort of closet, formed by a wooden partition reaching up to the roof of the building and down to the front of the top

of the pans. Opposite each pan is a shutter in the partition, which can be slid up whenever it is requisite to obtain access



FIGS. 4 AND 5.—RANGE OF BONE BOILERS.

to the pan. Each pan is closely covered with a wooden lid, and from the upper and back part of each pan, beneath the

cover, starts a pipe leading to a 10-in. main running the whole length above the pans and within the hopper, receiving contributions of vapour from each pan. This pipe finally has exit outside the building; it here communicates with an oblong condenser, made of sheet iron, filled with coke, measuring 14 ft. long, 3 ft. in vertical section, and 14 in. in horizontal section, inclined a little in its long diameter. At the lower end it receives the pipe from the boilers; and from its lower border another small pipe conveys away the water produced by the condensation of the vapour, the cooling agent being the outer air, to which the condenser is exposed on all sides. The upper end of the condenser communicates by two air-pipes with the flues from the fire-places. When the pan-lids are raised, the escaping vapour rises to the roof, and is conveyed from the interior of the closet into the chimney-shaft by escape-steam pipes provided for the purpose. The reference letters indicate: *a*, fire-holes; *b*, boiling-pans; *c*, steam-pipes; *d*, main steam-pipe; *e*, escape-steam pipes; *f*, air-pipes; *g*, chimney-shafts; *h*, condensed-steam pipes; *i*, water-trough; *k*, wrought-iron condenser; *l*, doors for cleaning smoke-flues; *m*, smoke-flues; *n*, closet with sliding door.

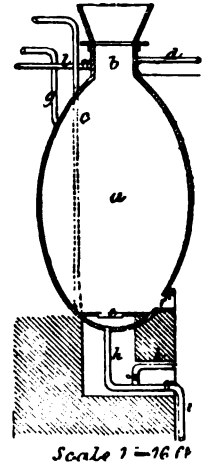


FIG. 6.—BONE DIGESTER.

The form of digester employed by Proctor and Ryland, of Birmingham, for extracting both fat and gelatin from bones, is shown, Fig. 6. It consists of a (duplicate) egg-shaped iron vessel *a*, capable of holding 6–7 tons of bones: at the upper end is a short neck *b*, surrounding the charging hole, which is closed by a tight-fitting cover; here enters the steam-pipe *c*, which descends to the bottom of the digester; here also the waste-steam pipe has its exit. At the bottom of the digester is a wooden false bottom *e*, for supporting the

bones, and on one side is a manhole *f* for reaching the false bottom and removing the boiled bones. Cold water can be introduced by the pipe *g*. The liquid matters are withdrawn by the pipe *h*, separating into 2 branches, with appropriate taps, the one *i* leading to a drain, the other *k* to a tank. The bones are first boiled in water by the admission of free steam; when sufficiently boiled, steam is turned off, and time is allowed for the fat to separate; cold water is then let in by *g* to raise the fat to the level of the pipe *l*, where it escapes into a receptacle. The water is next run off from the digester by the pipes *h i* into a drain, the top cover is fastened down again, and the bones are steamed at a pressure of about 50 lb. for the extraction of the gelatin; the gelatin is drawn off by the pipes *b k*, and the bones are finally removed by the manhole *f*. The bones are in a less friable condition than when the form shown in Fig. 3 is used, and they require to heat somewhat before being ground.

The product thus obtained varies in colour from drab to deep brown; it possesses a characteristic odour, and is liable to contain free fatty acids, as well as more or less lime dissolved in it (see Chap. IV.). The solidifying point of its fatty acids is usually about the same as in the case of lard, although the best specimens of bone tallow approach ordinary tallow in this respect. Bone grease must not be confounded with bone oil, which results from the decomposition of gelatinous tissue in bones, when they are calcined for the preparation of bone black, and the volatile products are condensed.

Kitchen and Ships' Grease.—The names of these sufficiently indicate the sources whence they are derived, and it is not an unusual thing for the soap manufacturer to purchase them in their rough form, as collected by the small dealers. The apparatus for, and mode of, rendering and purifying are the same as those described under the head of tallow and bone grease, but the operation is more offensive, and special pains should be taken to mitigate the nuisance

caused by the fumes.* The colour of the product varies as does that of bone grease, the older and sourer qualities of rough grease yielding a browner and inferior product. A well rendered sample should not contain any other impurity than about 0·5 per cent water, and its fatty acids should approach 108° F. (42° C.) in their solidifying point.

Glue and Skin Grease.—These greases are bye-products in the manufacture of glue, size, &c., and are usually very dark in colour, with a strong disagreeable odour, which does not disappear in the resultant soap. They are liable to contain water and sulphuric acid, as well as traces of gelatin, &c. Their fatty acids solidify at about 100° F. (38° C.).

Curriers' Grease is not unfrequently used in inferior soaps. It is a mixture of tallow and fish oils, and possesses the characteristic smell of the latter, combined with that of leather. The solidifying-point of its fatty acids varies with the proportion of fish oil used. Occasionally the variety known as "leather tallow" (which contains little or no fish oil) is met with, especially in imports from the United States.

Suds and soapy waters may be made to afford a small amount of material for soap-making by one of the following processes.

Tessié du Motay generates calcium, magnesium, or barium bicarbonates in the suds from cleansing wool, &c.; double decomposition ensues; the soluble salts are filtered off, and the insoluble are treated with a mineral acid, which isolates and permits the recovery of the resinous and fatty bodies.

Thorn and Stenhouse add calcium chloride to soap liquors obtained in clearing woven fabrics; the precipitate is filtered off and treated with hydrochloric acid, pressed, melted, and again pressed in a hot room, which removes most of the fat in a fit state for soap-making.

Benjamin treats wool-washing suds with boiling alum

* Some remarks under this head will be found in Chap. XI.

solution (2–3 cwt. alum to 8000–10,000 gal. suds); this is stirred, stood for 24–48 hours, and the liquid is drained off. The greasy mud is treated with hydrochloric acid to neutralise the alum; then hot-pressed in woollen bags. The extracted grease is refined by boiling with hydrochloric acid.

Neumann treats the waters from fulling mills, &c. with lime-water and iron or magnesium sulphate in suitable tanks. The iron or magnesia soap and the calcium sulphate resulting go to the bottom, leaving the water comparatively clean. The mud is repeatedly distilled to afford the fatty and other matters present.

Prévost treats the soapy water from wool-washing with a mixture of 1 part sulphuric acid at 168° Tw. (66° B.) and 3 parts at 116° Tw. (53° B.), and 1 part of hydrochloric acid at 36° Tw. (22° B.). A fatty magma floats, and is strained off; this is boiled with addition of some dry sawdust, and meanwhile constantly stirred.

Butter occasionally finds its way to the soap-maker, when it has become unsaleable for food purposes. It contains water and salt as impurities, and also the complex organic acids similar to those mentioned as occurring in rancid tallow. Although its fatty acids solidify at about the same point as those of lard, its value as a saponifiable material is not great, on account of the presence in it of several of the lower terms of the acetic series of acids (p. 8), which are themselves soluble in water, while their soda salts cannot be precipitated from their aqueous solution by the use of salt (sodium chloride). Butterine is also sometimes offered to the soap-maker; the basis of this is, or should be, the softer parts of tallow, the so-called oleo-margarin, whose fatty acids solidify at about 107° F. (42° C.).

Fish Oils of various kinds can only be used in the manufacture of soft soaps; they are usually offered for sale in a fairly pure condition, and require little or no previous preparation to fit them for the soap-pan. The chief caution to be observed in using them is that their smell is so pene-

trating as to hang about the vessels which are used to contain them, so that there is danger of contaminating other materials with their odour, unless special precautions are taken.

Further information about these fats and oils, and about some of the rarer kinds of fatty materials which are occasionally offered to the soap-maker, will be found under the head of "Oils" in 'Spons' Encyclopædia of Manufactures and Raw Materials,' at the end of which article also the bibliography of the subject is given.

SECTION B.—VEGETABLE OILS.

Palm Oil.—Although the consumption of palm oil by the soap-maker has enormously decreased of late years, in consequence of the comparatively low price of tallow, it is still an essential constituent of certain soaps, and it is one of the most important raw materials of the candle manufacture; a somewhat detailed description of it will therefore not be out of place in this volume.

Palm oil is the product of the outer fleshy coating of the fruit of several species of palm, but particularly of *Elæis guineensis* and *Elæis melanococca.*

For the production of commercial oil for exportation, the spadices are cut from the trees, and put in a heap in the outer air, where they are allowed to remain for 7–10 days; this causes the joints of the nuts to be weakened by the process of decomposition, and they are then easily detached by simply beating them. The nuts or fruits are gathered together, and the husks that adhere to their base are removed, either by hand or by rubbing them together, and are separated by throwing them in the air, and allowing a strong breeze to blow them away. A hole about 4 ft. deep is dug in the earth, and lined with plantain leaves, into which the nuts with their hard unyielding pulp are put and covered over, first with plantain leaves, and then with palm leaves and earth. The nuts are allowed to remain for

various periods—from 3 weeks to 3 months—until more or less decomposition has taken place, so that the pulp when removed is soft, and appears as if it had been thoroughly boiled. They are now put into a trough, made by digging a hole 4 ft. deep, and paving it below and around with rough stones. In some cases, a portion of the nuts is boiled in iron or earthenware pots, and then mixed with the unboiled portion before putting into the trough. They are now pounded with wooden pestles by men standing around the trough, until the pulp is quite detached from the surface of the hard nut; the whole is removed from the trough and piled in a heap, and the stones are taken out, leaving the oily fibrous pulp which is put into a pot with a small quantity of water over a good fire, and well stirred until the oil begins to melt out. The pulp is then put into a rough net, open at both ends, to which are attached 2 or 3 short sticks, by turning which in opposite directions the oil is squeezed out through the nets; it runs into a receiver or tub, leaving the fibre behind. The longer the nuts remain underground, the thicker the oil will be when made, the quality will also be inferior and the smell bad. On the other hand, the shorter the time, within certain limits, during which the nuts are underground, the better will be the oil made from them. It is evident from this rough mode of preparation that the oil is liable to contain more or less vegetable fibre, which is apt to act as a ferment, and render the oil rancid in course of time, besides holding in suspension a varying quantity of water. These impurities in the commercial oil are further increased by the fact that, when the oil is brought down to the coast, should no vessel be there prepared to receive it, it is frequently buried in the sand till an opportunity arrives for exporting it.

It has been estimated that the yield of oil from this palm is at the rate of 7 cwt. an acre, or more than $\frac{1}{3}$ greater than the oil product from the olive in Southern Europe. On some parts of the West Coast of Africa where the regular collection of the fruit is not practised, the trees grow so

thickly, and afford such regular and rapid crops, that the ground becomes covered with a thick deposit of the fatty matter afforded by the fallen nuts.

The oil obtained from the pericarp, the palm oil proper, has the consistence of butter, a yellow colour, an odour resembling violets, and a mild flavour; it easily becomes rancid, bleaches in the sunlight, saponifies readily with alkali, dissolves in all proportions of ether, and in alcohol of 0·848 sp. gr. Its specific gravity varies considerably, as also does the solidifying point of its fatty acids, which ranges from that of lard to above that of the hardest tallow. Besides its industrial applications to candles and soap it is largely used in the manufacture of tin-plate, in South Wales and elsewhere. For this latter purpose, its non-drying qualities render it valuable as a preservative of the surfaces of the heated iron sheet from oxidation, until the moment of dipping into the bath of melted tin, the sheets being rapidly transferred to that from the hot oil bath, which consists almost entirely of palm oil. The softest, purest and most neutral oil is preferred for this purpose, and hence the kind known as "Lagos" is much used.

Scarcely any oil which finds its way into commerce has a greater range of quality than palm oil. The various kinds are well known by the names of the parts of the coast whence they are shipped. The oil used to be brought home by small vessels trading from London, Liverpool and Bristol, which went out with empty casks, and lay some months along the coast, especially near the mouths of rivers, until they were filled up with oil. The great regularity with which steamers now call at many African ports, and the cheapness of freight, has materially altered the mode of conducting the trade. Casks are now left at the various depots, and instead of, as formerly, a 300-ton ship coming home laden with one or two classes of oil, steamers arrive regularly in Liverpool with cargoes chiefly of palm oil, made up at various ports of call, ranging perhaps from 8° N. and 13° W. (near Sierra Leone) to 10° S. and 12° E.

According to Lewkowitsch, the natural colouring principle of palm oil is of similar constitution to that of egg-yolk, and is destroyed by exposure to the air and by heat or chemicals, such as chromic acid, &c. Saponification by alkalies or lime does not destroy colour or odour, but acid saponification does both. The colour of the commercial brands ranges from pale yellow (Loanda), through ruddy orange (river oils), to dirty brown (Lahou). As palm oil is now never adulterated with other fat, such as palm-kernel oil, which would be fatal to its use for the production of stearine, the commercial valuation is confined to the determination of the water and insoluble matters, mostly sand. In the case of "Salt Pond" and "Grand Bassa" all impurities are allowed for by the seller. "Lagos" is bought *tale quale*, "as it is," but all other brands are purchased on an allowance for all impurities over 2 per cent. The following table presents, in concise form, the average characteristics of consignments arriving here from January 1890 up to July 1895 :—

Kind of Oil.	Number of Tests.	Water per cent. Average.	Solid Impurities per cent. Average.	Setting Point. Average.	Neutral Fat per cent.*
				°F. °C.	
Lagos	49	1·0	0·1	111·2 = 44·0	58–68
Bonny	120	3·6	1·3	111·5 = 44·2	44–88·5
Old Calabar ..	43	2·3	·8	111·4 = 44·1	76–83
Benin	83	1·9	·6	111·4 = 44·1	59–74
Cameroons ..	6	2·1	·4	111·2 = 44·0	67–83
Accra Addah ..	198	2·3	·3	110·5 = 43·6	18–76
New Calabar ..	20	4·2	2·4	111·8 = 44·33	40
Brass Niger ..	17	2·6	·7	111·7 = 44·28	35·5–47
Sherboro	7	2·7	·2	109·6 = 43·11	60–74
Salt Pond ..	21	8·9	1·9	115·2 = 46·22	15–25
Congo	..	3·9	·7	114·1 = 45·6	16–23
Lahou	..	6·0	·8	107·4 = 41·9	55–69
Half Jack ..	..	2·0	·2	105·4 = 40·8	55–57
Grand Bassa ..	..	3·4	·8	111·4 = 44·1	41–70
Popo	9	..	..	111·9 = 44·39	

* The neutral fat column is copied from Lewkowitsch.

For candle-making purposes the titer test is of utmost importance. The neutral fat column, as might be expected, shows great contrasts. In extreme cases the glycerides are entirely broken up, no neutral fat being left. Liverpool is now the only port in this country to which palm oil comes in quantity. The quantity for 1893 was 58,497 tons.

Palm-nut, Palm-kernel, or Palm-nut kernel Oil.—This oil, which is known in commerce under each of the synonyms given, is extracted from the nuts or kernels of the fruit described under Palm Oil. Some 20 years ago the nuts were partly charred, and the oil that ran from them, discoloured by the burnt cellulose, was exported either separately or mixed with palm oil. This brownish oil could only be very slightly bleached, and was therefore not of great value for soap-making. Since the improved methods of oil extraction, described later on, have been worked out, the nuts have been exported to England, and the nearly colourless oil there extracted, while the ground nuts have been used for cattle-food. The following is a description of the old process, which is still in vogue in some districts.

The nuts which have been subjected to the processes described for making oil, are deprived of their external pulp, and the kernels only remain; or old nuts are picked up from under the trees, and put in the sun for days, and even months, until they are perfectly dry. They are then broken between two stones, and the kernels are obtained whole, in perfect condition, and fit for exportation, and so form the commercial palm kernels. If they have not been perfectly dried, the kernels break into pieces. The oil obtained from these kernels by the following process is called "white-kernel oil." The kernels are placed in wooden mortars and pounded very fine; then removed to a grind-stone and ground into a homogeneous mass, which is put into cold water and stirred with the hand. The oil rises in white lumps on the surface of the water, and is collected and boiled. It is of a very light straw-colour, and, when

exposed to the sun and dew, becomes, after a time, perfectly white. "Brown" or "black" oil is thus obtained:—The kernels are put into a pan and fried; the oil oozes out, and is strained; the fried nuts are placed in wooden mortars, pounded, and afterwards finely ground on a grind-stone. The mass is thrown into a small quantity of boiling water and stirred constantly; the oil rises, and is continually skimmed off. The pulpy mass is removed from the fire, spread out in a large bowl, and allowed to cool, after which it is again ground, and put by until the cool of the day, when it is mixed with a little water to soften it. It is now beaten with the hand for some time until the oil comes out in white pellets. As soon as this is observed, a large quantity of water is put into it, and a fatty substance floats on the top; this is skimmed off and boiled, and the pure oil is obtained.

The greater portion, however, of the palm-kernel oil which is now used in commerce, is extracted from the nuts by the solvent action of carbon bisulphide. This plan is adopted on a very large scale by Heyl of Berlin and Sevin of London, and as the method is applicable to a great variety of purposes, it will be shortly described here. Olive oil residues, for example, treated in this way, yield a greenish oil much used for soap-making.

In the case of palm kernels, so completely is the meal freed from all traces of carbon bisulphide and its attendant nauseous-smelling compounds, that it is much used for cattle-food, though less valuable for that purpose than the meal from simple expression, by reason of its deficiency in oil. The residues from the extraction of olive oil in Southern Europe are dealt with in the same way, and made to yield an additional 2–4 per cent. of the so-called "pyrene" oil.

The method of employing carbon bisulphide may be illustrated by Figs. 7 and 8, showing respectively a longitudinal section and plan of Deitz's apparatus, It consists of extractors B, which are usually worked in couples alternately, and in connection with a still D, containing a steam coil, and

communicating with a cooler C by means of a pipe *e'*. The pipe *j* leads from the cooler worm to a receptacle A for

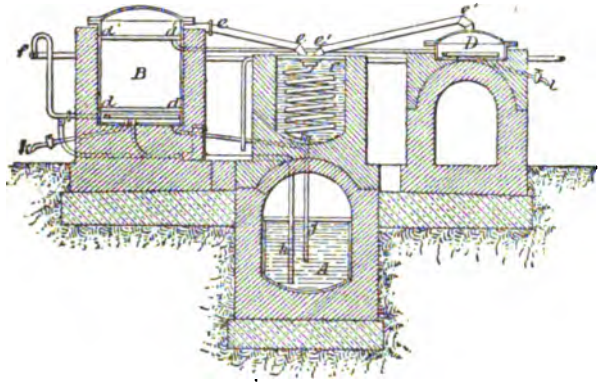


FIG. 7.—EXTRACTING OIL BY CARBON BISULPHIDE.

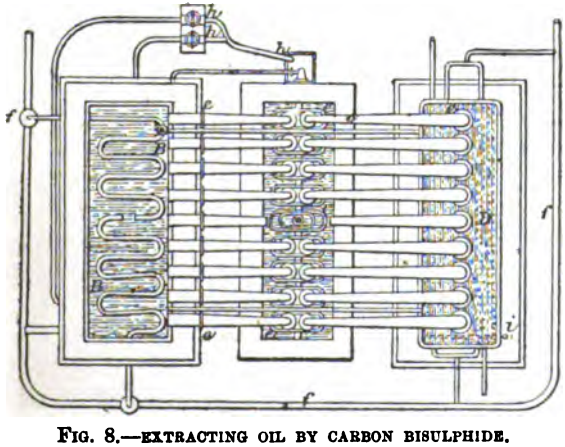


FIG. 8.—EXTRACTING OIL BY CARBON BISULPHIDE.

storing the bisulphide. The extractor B is also connected with the cooler C by the pipe *e*, and provided with suitable openings for the introduction and withdrawal of the fatty

matters. These latter are put into B between two perforated plates *d d'*; bisulphide is pumped from A by the pipe *h* in at the bottom of B. The bisulphide passes up through the mass, absorbs the fat present, and escapes finally by the pipe *f* to the still L. The admission of bisulphide is continued till a sample drawn off at *k* is free from fat. The bisulphide is then shut off, and steam is injected into the extractor through a perforated coil lying between the bottom and the perforated plate. The remaining bisulphide is thus carried by the pipe *f* into the receptacle A. The fat-saturated bisulphide is heated in D by means of a steam coil. The vapour of the bisulphide condenses in the cooler C, and runs back into A for further use. Steam is generally passed through the fat, to completely free it from bisulphide; and the clean fat is finally let out by the pipe *i*.

In America, preference is given to petroleum spirit of low boiling-point, as being cheaper and less dangerous, though less rapid in its action. The materials there principally dealt with are the residues from the rendering of animal fats (as tallow and lard), containing 12–15 per cent. of extractable grease. The patents relating to this industry in America are very numerous, but those taken out by Adamson seem to be most largely adopted. The process lasts 24–36 hours, and the products are used mainly as lubricants, being unfit for making good soap. "Pomace" or castor cake, and greasy cotton waste, are simply treated.

A very great disadvantage of petroleum spirit, however, is that whereas the high sp. gr. of carbon bisulphide enables it to be kept covered with water (and even collected from the mouths of the condenser worms under water), and thus protected from all risk of explosion by contact of its vapour with air and flame, petroleum spirit, which is, bulk for bulk, about half as heavy as the bisulphide, cannot be so protected from accident and loss by evaporation. Its vapour-density also is much less.

Other chemical solvents which have been proposed are sulphur dioxide and carbon tetrachloride. The use of the

former is patented by Grills and Schroeder, who employ it in a liquefied condition, at a temperature of 76°–104° F. (30°–40° C.), and under a pressure of 6 atmospheres. The application of the latter has been claimed by Lever and Scott, and it is said to afford a purer produ,t than can be obtained with carbon bisulphide.

When it is desired to produce a palm-kernel meal for feeding purposes, which shall not be totally devoid of fat, the palm kernels are treated by ordinary oil-mill machinery, one form of which will be briefly described under the head of cotton-seed oil.

By whichever of these processes it is obtained, palm-kernel oil, when freed from impurities, is of a pale primrose-yellow colour, with a characteristic odour not unlike that of coconut oil, which it strongly resembles in its chemical and physical characteristics. Indeed, for soap-making, it has largely supplanted coconut oil in the cheaper soaps in which that oil has hitherto been employed on a large scale. It is slightly softer than good coconut oil, its fusing-point being tolerably constant at 78° F. (25½° C.). If, however, the *whole* of the oil be removed from the kernels, the oil is slightly harder, containing a larger proportion of the higher terms of the series, lauric (or lauro-stearic) acid, cocinic acid, &c. The oil itself, being tolerably pure, is a neutral glyceride, and does not readily get rancid. Its fatty acids, however, are partly fixed and partly volatile, like those of coconut oil; 100 parts of it yield only 91·5 to 92 parts of solid fatty acids.

Olive Oil.—Olive culture is successful in the Old World wherever the mean annual temperature is from 58° to 66° F. (14½°–19° C.), that of the coldest month not falling below 42° F. (5½° C.), nor that of the hottest below 71° F. (22° C.). The *Olea europæa* seems to be the only species which is an object of systematic cultivation, and it thrives and is most prolific in dry, calcareous, schistous, sandy, and rocky ground, so that it may be grown on land which is worthless for many other crops. In the extraction of the oil there are

two distinct processes—crushing and pressing. In the first process the fruit is by some completely crushed; by others, the pericarp only is crushed first. The pulp or paste is shovelled into bags which are placed in the press (the latte being kept warm), and exposed to air as little as possible. "Virgin" oil is that obtained by the first pressing; the pulp or marc is then stirred up with boiling water, and pressed again, more strongly, giving an oil only slightly inferior to the first. The marc is again treated with boiling water, the stones are all thoroughly crushed, and the whole is again pressed, yielding what is known as "pyrene" oil. The husks and various oily crusts and residues are now frequently exhausted of their oil by the carbon bisulphide process described above, and as the oil thus obtained is richer in stearin than any other variety, it makes a harder soap.

Good olive oil is a somewhat viscid liquid, pale or greenish yellow, of faint, agreeable odour, and bland oleaginous flavour; sp. gr. 0·917 at 60° F. (15°·5 C.); it commences to lose transparency by separation of a crystalline fatty substance, chiefly stearin, at 32°–50° F. (0°–10° C.); when congealed and pressed, it affords ⅓ of solid fat melting at 68°–82½° F. (20°–28° C.), the fluid portion (olein) remaining liquid at 25°–14° F. (−4° to −10° C.) It is one of the less alterable, non-drying oils. The best kinds of oil are consumed enormously in food and medicine, constituting the "salad oil" of the shops; the commoner kinds are employed in lubricating, illuminating, woollen dressing, and the manufacture of soaps. It is most extensively adulterated with refined cotton-seed, ground-nut and other oils. We import some 20,000 to 30,000 tons of olive oil annually.

Coconut and Copra Oil.—As indicated by its name, this oil is obtained from the fruit of the coconut palm, *Cocos nucifera*, which flourishes with equal vigour on the coasts of the East Indies, throughout the islands of the tropical Pacific, and in the West Indies and tropical America. Ceylon, which is peculiarly favourable to it, is estimated to contain

some 20 million trees. The composition of the nut is said to be:—

Water	39·7	Emulsin	1·1
Oil	29·3	Albumen	0·5
Amygdalin	14·0	Mineral Matter ..	0·2
Woody fibre	9·5		
Sugar	3·6		100·0
Gum	2·1		

The albuminous pulp dried at ordinary temperatures (called "copra" or "copperah") contains 54·3 per cent. of oil, and dried at 212° F. (100° C.), 66 per cent. For preparing copra only ripe nuts should be collected, and they should not be broken till 4–6 weeks after gathering; the copra then dries more quickly, does not become mouldy, and affords a greater yield of oil.

All the native modes of extracting the oil are, comparatively speaking, rough and wasteful, but two of them may be briefly described here. The most effectual methods of extraction, however, are those described under the heads of palm-kernel and cotton-seed oils. Special disintegrators are necessary, the best form being made by Rose, Downs & Thompson, Hull.

An Indian process for extracting it, when required to be colourless for perfumery manufacture, is as follows:—The kernel is plunged into water and boiled for a few minutes, then grated and placed in a press; the emulsion thus obtained is boiled until the oil rises to the surface. This process is not cheap enough for ordinary commercial oils, and recourse is had to rude forms of oil-mill, worked by oxen, and treating about 130 lb. of copra daily, obtaining about 40 qt. of oil. Another plan is to divide the kernel into pieces and dry them on shelves over charcoal fires; after 2–3 days they are put into the press. By this method, 100 nuts carefully dried are estimated to yield by pressure 10–13 *edangalies* (of 92 cub. in.) of oil, or 40 nuts to a gallon; inferior nuts will not give more than 3–9 *edangalies*; those from trees on salt marshes afford the least oil. Recently a

factory has been erected at Borongan for the better preparation of the oil. The grating of the pulp is performed by iron discs with toothed edges, radiating from the ends of iron rods, and bluntly pointed towards the centre of the fruit. These discs are made to rotate by suitable gearing, while the workmen force the inner face of half cocoanuts, held firmly by both hands, and pressed by means of a pad on the chest, against the revolving rasps. The finely shredded nut lies for 12 hours in flat pans, to undergo partial decomposition, and is then gently pressed; the resulting liquor, consisting of $\frac{1}{3}$ oil and $\frac{2}{3}$ water, is caught in tubs, and after standing for 6 hours, the supernatant oil is skimmed off. The latter is next heated in iron pans, holding about 20–25 gal., until all the water has evaporated, occupying 2–3 hours. In order to cool the oil rapidly, and prevent its deepening in colour, 2 pailfuls of cold oil, freed from water, are poured in, and the fire is quickly withdrawn. The compressed shreds are once more exposed to the air, and then subjected to powerful pressure. After these two operations have been twice repeated, the rasped substance is suspended in sacks between two strong vertical boards, and alternately squeezed and shaken up for a considerable time. The refuse finally serves as pig-food. The oil which runs from the sacks is quite free from water, and very clear, and is used for cooling that extracted by the boiling process.

When pure, coconut oil is white, and almost as fluid and limpid as water, in tropical climates, but solidifies at 61°–64½° F. (16°–18° C.), and consequently generally appears in Europe as a solid, opaque, unctuous substance, of the consistence of butter, and fusible at 75°–82° F. (24°–28° C.). When fresh, its odour and flavour are sweet and agreeable, but it quickly becomes rancid. It dissolves readily in alcohol, and saponifies with facility. It contains at least ten different fatty acids, of which the chief is laurostearic, together with oleic, palmitic, myristic, and some of less importance, all combined with glycerin. In Europe, the chief applications of the oil are for candle and soap making. It is an excellent illu-

minator, in both candles and lamps, as it emits no smoke; and it forms a hard and very white soap, more soluble in salt water than any other kind made on a commercial scale. During the last 25 years its consumption for soap-making in England has been greatly reduced by the substitution of palm-kernel oil, extracted here. In the East, the oil is largely used for cooking and medicine while fresh, and for burning, painting, soap-making, and anointing the body, when rancid.

It has been already noticed that coconut and palm-kernel oils combine with a larger quantity of soda than any other known fat, and hence that the yield of soap from these oils is greater than from other fats. Further, the soap so produced has the power of combining (and making a hard soap) with more water than can ever be communicated to tallow soap, a property which has frequently given rise to dishonest traffic. This soap is more soluble in water than any other, and requires a much larger quantity of common salt to separate the excess of water from it. In technical language, it is said to "work close." Reference to the table on p. 8 will show the reason for this. Coconut oil contains a number of low terms of the fatty-acid series, whose salts are so very soluble that even the lime and baryta salts of the lower among them are quite soluble in water, just as are the lime and baryta salts of formic and acetic acids, the lowest terms of all. Our annual imports of coconut oil range between 6500 and 15,500 tons.

Castor Oil.—The inferior qualities of this oil are not unfrequently found among soap-making materials. The plant, *Ricinus communis*, or *Palma-Christi* as it is often called, is indigenous to India, whence it has been distributed by cultivation through the tropics and in many temperate countries. There are two varieties of it, distinguished by the size of their seeds; the small-seeded variety yields the better oil. The medicinal variety is expressed from the seeds without any heat.

The manufacture of castor oil is actively carried on in

the United States, especially at St. Louis, the seeds being largely produced in S. Illinois. Kansas and other states participate in the industry. The land is prepared as for other crops, and the seeds are planted much the same as maize, except that only one seed is put into each hill, and that every fourth row is missed to afford space for the harvesting. Ripening commences in August; the yield varies from 15 to 25 bush. an acre, 20 being considered fair. The oil is generally extracted in the following manner:—The seeds, having been thoroughly cleansed from the dust and particles of the pod with which they are always contaminated, are subjected to decortication, the most efficient machine for the purpose being that made by Rose, Downs & Thompson, Hull. Next they are placed in an iron tank, and heated to such a degree as will liquefy the oil without any risk of scorching. They are then pressed, the oil escaping being known as "1st quality." The pressed seed is heaped up and left for a day; on the following day it is again heated and pressed, and gives a "2nd quality" oil. The yield from 1 bush. of seed is 12 lb. of 1st quality, and 4 lb. of 2nd quality oil; sometimes a third expression is made, giving 1–3 lb. of a very highly coloured oil. Occasionally, too, the cake from the second pressing is treated with carbon bisulphide, which extracts a small additional quantity of thick, dark, common oil. All qualities need purifying and clarifying. This is usually effected by boiling first with a large quantity of water, and skimming off the impurities as they rise, while the mucilage and starch are dissolved, and the albumen is coagulated. The clear oil is removed and boiled with a very little water, which clarifies it and drives off volatile acid matters. The chief point to be noticed is to expose the oil as little as possible to the air, or it quickly becomes rancid. The 1st quality oil is used medicinally, the 2nd for burning, lubricating, leather dressing, &c. In America the cake is frequently used as fuel.

Castor oil has a sp. gr. of 0·960–·966 at 60° F. (15°·5 C.), 0·957 at 77° F. (25° C.), and 0·908 at 201¼° F. (94° C.); it

has usually a pale yellow colour, viscid consistence, and slight mawkish odour and flavour, which are much intensified when it becomes rancid; at 5° F. (– 15° C.) it commences to congeal, but does not completely solidify above 0° F. (– 18° C.), when it dries up, forming a transparent varnish-like film in three layers; its boiling-point is 509° F. (265° C.), when it begins to distil, and affords various products; it mixes in all proportions with glacial acetic acid and absolute alcohol, and is even soluble in 4 parts alcohol of 0·838–0·850 at 59° F. (15° C.), and forms a clear mixture with equal weights of the same at 77° F. (25° C.); it is said also to render other oils mixed with it soluble in alcohol. It saponifies readily, yielding several fatty acids, the chief of which is ricinoleic ($C_{18}H_{34}O_3$), peculiar to this oil, and another appears to be palmitic. Its drastic principle and optical properties still await investigation. Some of its uses have already been alluded to. For medicinal use, as a purgative, the cold-drawn oil is the only kind fit for human subjects; but the oil obtained by roasting and boiling, or that extracted by alcohol, is preferred by veterinary surgeons, as containing much more of the drastic principle, and being therefore more powerful. The common kinds are largely used by leather dressers, principally, perhaps, for morocco leather, but with equal success on all descriptions; moreover it repels rats and other vermin, and does not interfere with subsequent polishing. As a lubricant, it is in extensive use in Europe and America; and as a lamp oil, in India, Brazil, &c. Chiefly 3 sorts appear in the London market—that expressed here from imported (essentially Egyptian) seed, E. Indian expressed, and American expressed. The oil is imported in tins, barrels, hogsheads, and duppers. It is often adulterated with poppy-seed oil and croton oil.

Cotton-seed Oil.—The seeds which are separated from the "lint" or "wool" of the various kinds of cotton in the process of "ginning" are valued for their oil. The utilisation of this oil has of late years assumed the importance of a distinct industry in the United States, where there are now

upwards of 40 oil mills, 9 being situated in Mississippi, 9 in Louisiana, 8 in Tennessee, 6 in Texas, 4 in Arkansas, 2 in Missouri, 2 in Alabama, and 1 in Georgia. The quantity of seed there treated for its oil now amounts to over 400,000 tons annually. The American process of extraction is as follows:—The seed coming from the ginning operation still has some fibre adhering to it, and has a tendency to accumulate in masses. These are thrown into a machine containing a screw-knife revolving in a trough, which divides the materials into particles fit for the screening operation. This is conducted first in a sieve with meshes that allow the sand and dirt to pass, while retaining the seed; and then in one through which the seed can escape, but not husks and coarse foreign matters. The cleaned seed is next passed through a special gin for removing all remaining fibre (useful for paper making and other purposes); and finally through a hulling machine or decorticator, consisting of fixed and revolving knives set so close as to sever the seeds. The best machine for the purpose is that made by Rose, Downs & Thomson, Hull, which is efficient even on very badly ginned seed.

The seeds, when hulled, are taken through one or more separators, which pass the kernels but retain the shells. The kernels are pressed into cakes between iron rolls, and are then placed in steam-jacketed iron tanks, 4 ft. wide and 15 in. deep, where, by constant stirring, and the action of dry heat obtained from injecting steam at 35 lbs. a sq. in. into the jacket, the oil is liberated from the cells in the course of about 5 minutes. The heated mass is then filled into sacks and subjected to repeated hydraulic pressure, till most of the oil is extracted. By this plan, the yield from 1000 lb. of seed averages 490 lb. of husks, 10 lb. of cotton, 365 lb. of cake, and 135 lb. of oil. Its value in New York is about 18–20*d.* a gal. The cake remaining after the expression of the oil is invaluable as a cattle-food and as a fertiliser, and is an important article of commerce.

A chemical process for decorticating cotton seed has been introduced of late years in the United States by Dudley and

Perry. The lint-freed seeds are submitted to the action of nitrous anhydride and sulphur dioxide, accompanied by air sufficient to regenerate the higher oxides of nitrogen as fast as they are reduced. The action on the fibre is said to be very rapid and complete, leaving the seeds smooth and clean, and the small traces of acid remaining on their surface can be readily washed off. But the introduction of such highly poisonous and corrosive substances into an oil mill is not likely to be tolerated, on account of risks both to workmen and machinery, to say nothing of possible injury to the product by carelessness or oversight.

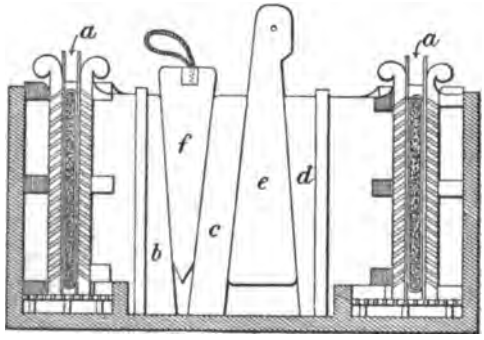


FIG. 9.—WEDGE PRESS FOR OIL SEEDS.

In India, cotton seed is used more as a cattle food direct, than as an oil yielder. By the native mills it affords 25 per cent. of a good oil, which, if purified, might become of considerable commercial importance. The seed cannot safely be shipped without undergoing preliminary cleansing, otherwise it heats and deteriorates in bulk on the long voyage. Egypt exports large quantities of the seed to English ports, and smaller amounts to Mediterranean ports.

According to Leopold Field, there were 3,000,000 tons of cotton seed produced in 1882, of which only 300,000 tons were worked up, meaning 90,000,000 gal. of oil lost.

To describe all the various methods adopted for the

extraction of oil from oleaginous nuts and seeds would be beyond the scope of this volume; a detailed account of oil-mill machinery may be found in such works as 'Spons' Dictionary of Engineering.' The most important only can be dealt with here.

But before describing modern improved pressing apparatus, reference must be made to the old-fashioned wedge press, which is still widely used in countries where modern engineering skill has not yet gained ground. Fig. 9 illustrates the lower portion of such a machine, where the

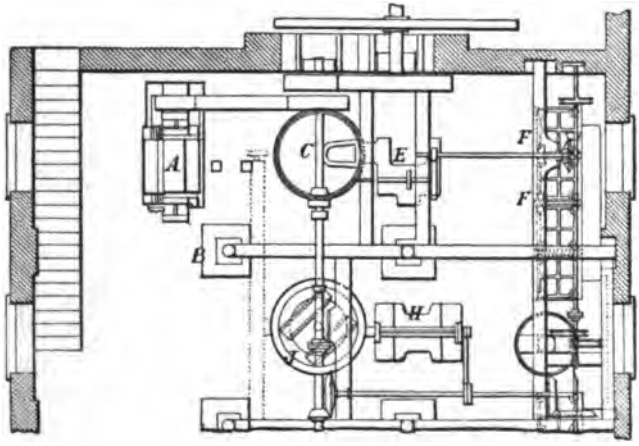


FIG. 10.—PLAN OF SEED OIL MILL.

expression actually takes place. The seed bags *a* are enclosed between perforated wooden boards, which deliver the outflowing oil to receptacles beneath. The slabs *b c d* are wooden counter wedges; the tightening wedge *e* is forcibly drawn up, while *f* is driven down by a "monkey," the whole being fixed in a frame with ropes and levers after the manner of a pile-driver.

Probably the best system is that introduced by Rose, Downs & Thomson, of Hull, which is the chief seat of the seed-oil industry in England; it will now be described.

The arrangement of the mill is shown in plan in Fig. 10, and in elevation in Fig. 11. The seed or other material passes through the following course:—It runs from an upper floor through the roll-frame A, by which it is crushed 3 or 4 times; it is then taken by the elevators B to the kettle C, where it is heated and damped. From beneath the kettle it is drawn, in quantities sufficient to make a cake, by a box which conveys it to the moulding machine E. Here it undergoes preliminary compression, the objects of which are (1) to increase the number of cakes which may be inserted in the

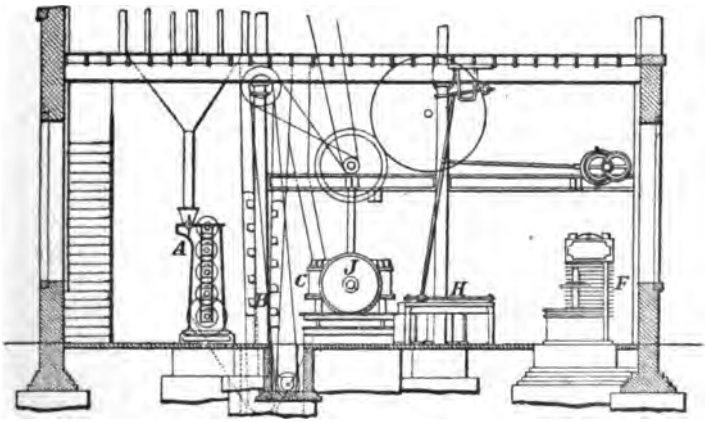


FIG. 11.—ELEVATION OF SEED OIL MILL.

presses at one time, enabling 18 12-lb. cakes to be made where 4 8-lb. cakes were formerly made, and (2) to ensure uniform size and weight, and uniform density or consistence throughout. The cakes are removed from the moulding machine, and put into the press F, 3 or 4 of which are required to each moulding machine. The pressure is applied either by means of hydraulic pumps, or by a high-and-low-pressure accumulator; but unless extreme care is used with the latter, it gives too rapid a pressure, squirting out the seed at the side of the plates and exercising a destructive

effect upon the cloth employed. The pulsation caused by the pumps working directly to the press cylinder is more akin to the action of a wedge, and seems to extract the oil better than the dead pressure given by the accumulator. If the latter is used, a small cylinder may be applied to give the preliminary pressure in the moulding machine, in lieu of a cam. After remaining under pressure about 25 minutes, the cakes are withdrawn, and after being stripped of the cloth,

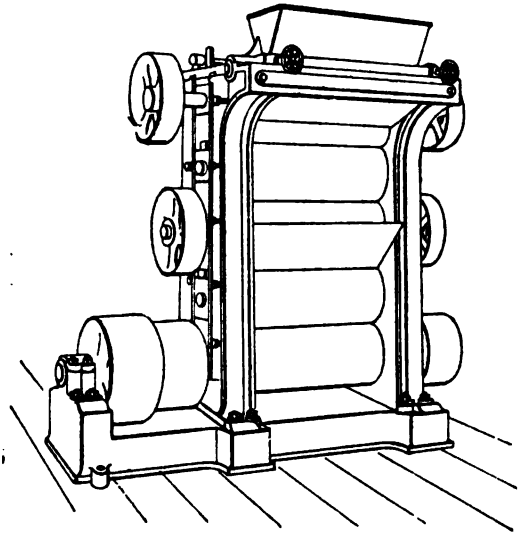


FIG. 12.—OIL-SEED ROLL-FRAME.

are pared by the machine H, which completes the manufacture of the cakes. The parings fall under a very small pair of edge-running stones J, which automatically discharge them, when sufficiently ground, into an elevator conducting to the kettle, where they are worked up with fresh seed. In a mill with 4 presses, 2 men and a boy in the press-room can make 6 tons of cake in 11 hours, a rate of production requiring 6 men by the old process. The saving in steam-power is

about 30 per cent., chiefly due to the absence of the heavy edge-runners, which also effects an economy of space. About 2 per cent. more oil is extracted, and the cakes are improved in appearance by not having the structureless texture caused by the trituration of the seed under edge-runners.

The general routine of the process having been described, details may be added concerning the working of the several machines. The roll-frame, Fig. 12, consists of 4 or 5 chilled-iron rolls, each 3 ft. 6 in. long by 16 in. diameter, placed one above the other. These rolls are used for crushing all the seed that passes through one set of presses, making 5½–6¼ tons linseed cake per spell of 11 hours. The seed passes into the hopper in the usual manner, and is distributed to the crushing rolls by a fluted feed-roll the same length as the crushing rolls, placed at the bottom of the hopper. When the seed passes the feed-roll, it falls on a guide-plate that carries it between the 1st and 2nd rolls. After passing between these rolls and being partly crushed, it falls on a guide-plate on the other side, which carries it back between the 2nd and 3rd rolls, where it is crushed more fully. It then falls on another guide-plate, which carries it between the 3rd and 4th rolls, where it is ground more fully; then it falls on a 4th guide-plate, and is conveyed between the 4th and 5th rolls to receive the finishing touch. It is thus crushed 4 times.

The kettle is shown in Fig. 13, which represents one capable of heating sufficient seed to keep 4 16-plate presses occupied, or to make 6 tons of cake per 11 hours. It is steam-jacketed and furnished inside with a damping apparatus. The inside diameter is 5 ft. and the depth 2 ft. 6 in. The seed introduced is kept in motion by the stirring-gear, and when sufficiently heated and damped, is withdrawn by the box A in quantities to form one cake, and transferred at once to the moulding machine, attached or separate.

This machine is illustrated in Figs. 14, 15. Its purpose is to measure the quantity of seed required to make each cake, to shape it as required, and to press it so much,

without extracting any oil, as will enable the greatest number of cakes to be put into the press. The measure of seed is placed on a strip of woollen cloth, spread upon a thin iron tray, sliding on the guides B; the bottomless hinged mould C having the exact shape of the intended cake, is closed upon it, and the measure A (Fig. 13), which is also bottomless, is drawn over guides in the upper surface of the mould C, thus accurately distributing the seed. The mould

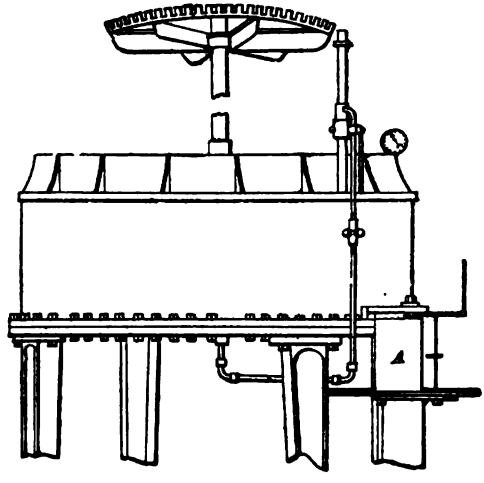


FIG. 13.—OIL-SEED KETTLE.

is next thrown upon its hinge (Fig. 14), and the ends of the strip of cloth are folded over the seed, the thickness of which is about 3½ in. The thin iron tray, with the mould of seed upon it, is then pushed along the guides B, beneath the die D. This action gives motion to a cam, shown above in the illustrations, but which may be placed beneath if necessary. This cam brings down the die, and compresses the mould of seed to a thickness of 1¼ in.; its revolutions are so timed that the seed is under pressure long enough (about ⅓ minute) to let the workman have another cake ready.

When the die of the moulding machine rises, the cake and tray are removed and placed in the press (Fig. 16), the tray being withdrawn. The plates of the press are slightly

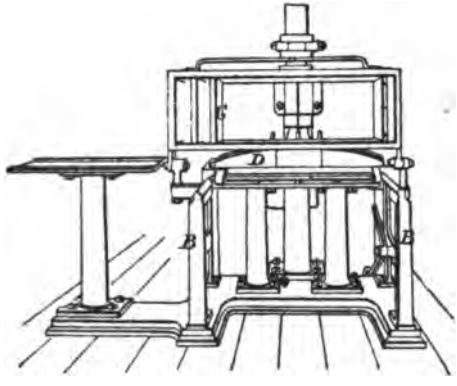


FIG. 14.—SEED-CAKE MOULDING MACHINE.

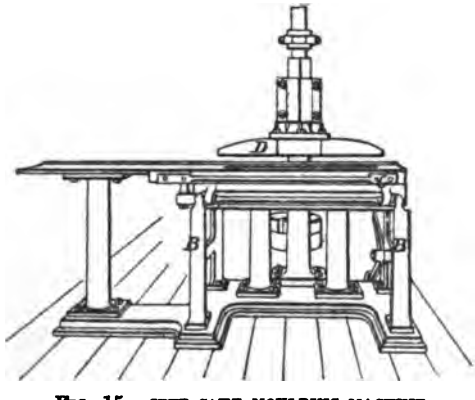


FIG. 15.—SEED-CAKE MOULDING MACHINE.

thickened towards the edges, and bear the name of the manufacturer in reverse. The press is suitable for extracting oil from linseed, rapeseed, cotton-seed, hempseed, niger-seed, sunflower-seed, gingelly-seed, castor-seed, ground-nuts,

coconuts, olives, &c. It is made in various sizes. The No. 1 double press (not shown) is furnished with 4 cake-boxes, suitable for making 4 tapered cakes at one pressing, each about 2 ft. 5 in. long, by 10½ in. wide at one end, and 7½ in. at the other, when using linseed, 48 lb. of Bombay seed being required to charge the press, and giving a cake weighing about 8 lb.; the maximum and minimum weights of its charges are 60 lb. and 40 lb.; of the cakes, 13 lb. and 6½ lb. The charges vary from 3 to 6 an hour, being 4 for cotton-seed and 5 for linseed; most other seeds are worked the same as linseed, but rape and gingelly are worked twice. By using 2 presses for the first time and 3 for the second, 3 presses will crush as much seed as 5. These presses are made of a capacity to take 270–320 lb. seed at a charge, giving cakes of 9–15 lb., and requiring 30–45 minutes for the operation. In all these presses, the hair wrappers, weighing some 26 lb., used in the old process, are dispensed with.

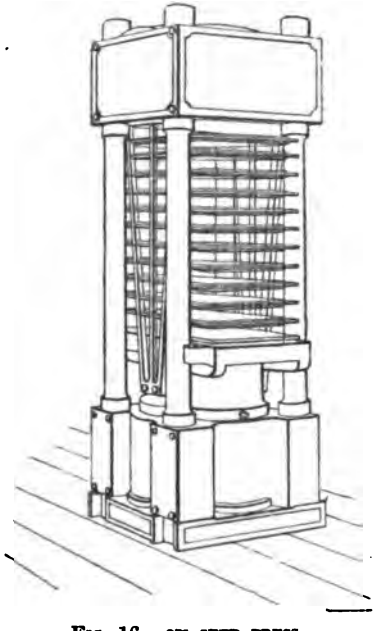


FIG. 16.—OIL-SEED PRESS.

After being sufficiently pressed, the cakes are withdrawn, stripped of their cloths, and pared by the machine shown in Fig. 17, which consists of 2 reciprocating knives moving above a table fitted with guides and gauges, and with a

screw conveyor in the centre, for discharging the parings at one end. Two boys attending the machine can pare 12–13 tons a day. The parings are taken under a very small set of edge-runner mill-stones, which automatically discharge them, when sufficiently ground, into an elevator leading to the kettle, where they are worked up with fresh seed. Larger edge-runners are very commonly used for crushing Egyptian cotton-seed; a set weighing 404 cwt. crush about 6 tons per 11 hours.

The ordinary method of liberating the cocoanut from the shell is to break the latter into 2 or more pieces by blows from a hammer, and to leave these exposed to the sun's rays

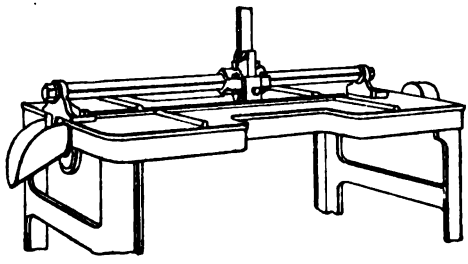


FIG. 17.—SEED-CAKE PARING MACHINE.

for a few days, when the kernel contracts, and leaves the shell. Some years since, Rose, Downs & Thompson designed 2 machines for slicing and rasping the dried kernel, or copra, which, in general construction, resembled gigantic mincing machines. The slicing machine worked well, and turned out a large quantity, but it was found to be of little value in practice, on account of the frequency with which stones and similar foreign bodies got mixed among the copra, and injured the knives. The rasping machine also suffered in a minor degree from the same cause. Moreover, there was no real gain in using the machines, as they did not reduce the material to a sufficiently fine degree. The

first produced thin slices, and the second rendered these granular only, like coarse sawdust, so that it was still necessary to pass the material through an edge-runner mill, and no perceptible difference was made in the amount of grinding required in this latter. Consequently, both machines have been abandoned, and the broken copra is thrown at once under edge-runners, and then pressed and dealt with much in the same manner as an ordinary oleaginous seed. The firm named estimate the average cost of the machinery adapted to a small mill capable of treating about 40 cwt. of copra daily, and making 24–25 cwt. of oil, and 15–16 cwt. of cake, as follows:—Engine, boiler, edge-runners, presses, pumps, gauges, "hairs," cisterns, pipes, nuts, bags, bagging, and all fittings complete, at about 1000*l.* The same motive power will drive also the machinery for preparing the fibre from the husk, costing about 150*l.* additional. These figures are, of course, subject to some fluctuation. Moreover, improvements in one or another branch of the industry are being constantly introduced, to meet the varying conditions of different countries, and the requirements of new oil-yielding substances, which it is impossible for any text-book to keep absolutely informed on; therefore, those who are or may be interested in the subject should put themselves in communication with Rose, Downs & Thompson, of Hull, who make a special study of this trade, and who are almost exclusively responsible for the advances which have lately been made in it.

The yield of oil varies with the season and the locality in which the seed is produced. In the United States, it is reckoned that for each 1 lb. of ginned cotton there are 3 lb. of seed, or a total approaching 4000 million lb., half of which only is required for sowing. One authority estimates that 100 lb. of seed give 2 gal. of oil, 48 lb. of oil-cake, and 6 lb. refuse fit for soap-making; another says that 1 ton of American seed gives 20 gal. of oil; a third, that the oil product is 37 per cent. of the weight of the kernels; a fourth, that 2 gal. of oil and 96 lb. of cake are afforded by 1 cwt. of

seed; a fifth, that 1 ton of cotton material yields at the rate of some 35 gal. of oil.

The crude oil from new seed is clear and of a claret colour, but that from old or heated seed is opaque and much darker in colour. When refined it much resembles yellow olive oil, and has a pleasant, sweetish flavour; sp. gr., 0·920–0·927 at 60° F. (15°·5 C.); congealing-point, 34° F. (1° C.); it is largely used in adulterating other oils, as linseed oil, sperm oil and lard oil, for painting, burning in lamps and lubricating, but for this purpose its tendency to become "gummy," i. e. to "dry," or oxidise, is objectionable. It is extensively mixed with olive oil, and often replaces it altogether; as a preventive measure, the Italian Government has levied a heavy duty on its importation. Large quantities of it are consumed by soap-makers, in combination with other oils and fats. For lubricating purposes, some manufacturers prepare a "winter oil" from it, which does not thicken in cold weather, by precipitating and removing the stearin; but its gumminess must limit its application. The stearin so removed is usually made into soap. Although the oil itself is liquid at ordinary temperatures, the solidifying point of its fatty acid is about 98°–100° F. (37°–38° C.), and it enters very largely into the composition of the lower qualities of blue mottled soaps.

Linseed Oil. Almost the whole of the flax now grown is produced from the *Linum usitatissimum*, and after the crop is harvested, the first operation is the removal of the seeds by a mechanical process termed "rippling."

The supplies of linseed for crushing are furnished chiefly by Russia and India. It is found that, as a general rule, the colder the climate in which the seed is grown, the greater are the drying properties of the oil, but the worse is its colour. In India, preference is given to white seed, as yielding 2 per cent. more oil, affording it more freely, and giving a softer and sweeter cake, than the red seed; the latter, moreover, always comes to market largely mixed with rape-seed, which is very difficult of separation, and greatly

depreciates the market value. Oil from unripe seed is watery. The seed should always be kept for 3–4 months in a dry place, as the oil furnished after this lapse of time is much more abundant than when the expression takes place immediately after the harvest. The seed is crushed and pressed in the manner described above. The best and finest oil is that which is "cold-drawn"; it is paler, less odorous, and less flavoured, but the yield is only 21–22 per cent. of the seed. By the aid of a temperature not exceeding 200° F. (93½° C.), and powerful and long-continued pressure, as much as 28 per cent. of very good oil can be obtained. The cake forms a valuable cattle food. The Italian variety is said to have a much more highly oleaginous seed than the Russian.

Linseed oil has a faint colour, and mild odour and flavour when pure, but the commercial article is dark yellow, with sharp repulsive flavour and odour. Its sp. gr. at 60° F. (15°·5 C.) is 0·934; at −18° C. a little solid fat separates out; at 4° F. (− 20° C.) it solidifies. By exposure to the air, after heating with lead oxide, it rapidly dries up to a transparent varnish. The fresh oil saponifies readily, giving a yellow and very soft soap with soda; by saponification, it yields 95 per cent. of fatty acids, chiefly linoleic, with a little oleic, palmitic, and myristic acids. It dissolves in 1·6 parts of ether, and in 32 parts of alcohol at 0·820 sp. gr. The oil is very extensively used in the manufacture of paint, printing-ink, floorcloth, artificial rubber, oil-varnishes, and soft soap. For artists' use, it is purified by shaking up with whiting, and warming. Linseed oil is never met with in commerce really pure, nor even is the seed itself. Previous to the Crimean War, it was a recognised custom at the Black Sea ports to add 1 measure of hemp or other seed to every 39 of linseed. Since then the proportion has advanced to 1 in 19, in addition to which, the Indian seed is grown mostly as a mixed crop with mustard and colza; pure linseed-oil can only be obtained by picking out the seeds individually.

Hempseed Oil.—The seeds of the hemp-plant so well known as a fibre-producer, are valued for their oil. It

is from Russia and Lorraine that the seed for expressing mostly comes. When the fibrous stems are tied in bundles the seed is rudely threshed out, and spread in thin layers under cover to dry. The extraction of the oil is performed in the same manner as with other seed oils. The proportion of oil contained in the seed is about 34 per cent. on an average; the yield varies from 25 to 30 per cent. The oil is at first greenish or brownish yellow, deepening with exposure to the air; the flavour is disagreeable, and the odour is mild. It has a sp. gr. of 0·9252 at 60° F. (15°·5 C.); it thickens at 5° F. (– 15° C.), and solidifies at – 13° to – 18° F. (– 25° to – 27½° C.); it dissolves in 30 parts of cold alcohol and any proportion of boiling; it saponifies with difficulty, forming a soft soap, but less soft than that from linseed oil. It is locally consumed largely for lighting, but its most important application is for making soft soaps.

Colza and Rape Oil are identical, and are derived from the seed of several species or varieties of *Brassica*, chiefly *B. campestris* and *B. Napus* (*Napa oleifera*), cultivated chiefly on the Continent and in India.

The oil is extracted from the seed by the usual methods; its sp. gr. at 60° F. (15°·5 C.) is 0·912 to 0·920, and it congeals at 21° F. (– 6° C.). Its colour is brownish yellow, and it is chiefly used for illuminating and lubricating purposes, and in lesser quantities for making soft soaps.

Gingelly, Sesame, Til, or Benne Oil.—Gingelly oil, whose name is variously spelt, is obtained from the seeds of *Sesamum indicum* [*orientale*], an annual plant 2–4 ft. high, indigenous to India, but long since propagated by cultivation in almost all tropical and sub-tropical countries, and now found nowhere in the wild state. There appears to be only one true species, but Indian cultivators distinguish two varieties—a white-seeded, called *suffed-til*; and a black-seeded, called *kala-til*.

In France, where this oil is very largely prepared, it is usual to subject the Levant seed, which is considered the best, to 3 processes of expression. After the first simple

expression, affording superfine (*surfine*) oil, the cakes are softened with cold water, and again pressed (*pression à froid* or *froissage*), and finally they are treated with steam and hot water for a third pressing (*pression à chaud* or *rabat*). The average product from 100 lb. of seed is 30 lb. of oil by the 1st pressure, 10 lb. by the 2nd, and 10 lb. by the 3rd. Calcutta seed gives 47 lb.: 36 lb. by the *pression à froid*, and 11 lb. by the *pression à chaud*. Bombay seed affords also 47 lb.: 25 lb. superfine, 11 lb. by the *pression à froid*, and 11 lb. by the *pression à chaud*. This last is contrary to the experience of Dantzig seed-crushers, as mentioned previously. The commercial oil has a sp. gr. of 0·923 at 60° F. (15°·5 C.), and some extracted by ether, 0·919 at 73½° F. (23° C.). This latter solidified at 41° F. (5° C.), becoming turbid at several degrees above this point; yet the congealing-point of the ordinary oil is placed at 32° F. (0° C.) by Prof. A. B. Prescott, and at 23° F. (– 5° C.) by Dr. Pohl. Visible ebullition commences at 212° F. (100° C.). At ordinary temperatures, it is more fluid than ground-nut oil, and is less liable to change under the influence of the air; indeed, when well prepared, it is said to keep for years without manifesting any rancidity. The oil is essentially composed of olein, which is sometimes present to the extent of 76 per cent., but it is not invariable in commercial samples. Among the other fatty acids, are stearic, palmitic, and myristic. The oil is frequently adulterated with ground-nut oil. It is said that 10 per cent. of gingelly oil in admixture with other oils may be detected by shaking 1 *grm.* of the oil with 1 *grm.* of a mixture of sulphuric and nitric acids previously cooled, when a fine green colour is produced which no other oil exhibits. The local uses of gingelly oil are for cooking, medicine, anointing the body and hair, absorbing the fugitive odours of plants, and illumination. In Europe, the superfine quality largely replaces olive oil for domestic purposes, and the other grades are employed by soap-makers.

Ground-nut, or Arachis Oil.—The ground-nut or peanut, *Arachis hypogæa*, is very widely cultivated in all

tropical and sub-tropical countries, for the sake of its oily seeds. Of the American crop, nearly one-half is grown in Tennessee. In Java, the oil is extracted by drying the seeds in the sun, and then subjecting them to pressure. In European mills, the nuts are first cleaned, then decorticated and winnowed, by which the kernels are left perfectly clean. An excellent decorticator for ground-nuts is made by Rose, Downs & Thompson, Hull. The kernels are crushed like any other oil-seed, and put into bags, which are introduced into cold presses; the expressed oil is refined by passing through filter-bags. The residual cake is ground very fine, and pressed under 3 tons to the inch, in the presence of steam heat; this affords a second quantity of oil, inferior in quality to the cold-pressed. The usual product is 1 gal. of oil from 1 bush. of nuts by the cold process, beside the extra yield by the hot-pressing. In France, where the oil is most largely prepared, 3 expressions are adopted, as with some sorts of gingelly: the first gives about 18 per cent. of superfine oil, fit for alimentary purposes; the second, after moistening with cold water, affords 6 per cent. of a fine oil, suitable for lighting and for woollen dressing; the third, after treating with hot water, yields 6 per cent. of *rabat*, or oil applicable only to soap-making. In India, the total mean yield is 37 per cent. at Pondicherry, and 43 in Madras. The cold-pressed oil is almost colourless, of agreeable faint odour, and bland olive-like flavour. The best has a sp. gr. of about 0·917 at 60° F. (15°·5 C.); it becomes turbid at 37½° F. (3° C.), concretes at 26½°–25° F. (−3° to −4° C.), and hardens at 19½° F. (−7° C.). By exposure, it changes very slowly, but thickens with time, and assumes a rancid odour and flavour. As an illuminating oil, it has feeble power, and its chief industrial uses are for soap-making and lubricating, particularly the former. Locally it is employed for cooking and burning, and as a general substitute for olive oil. Indeed, very large quantities are readily passed off as olive oil in European markets. As a rule, the seeds are exported in a raw state to such centres as London,

Marseilles, Bordeaux, Nantes, Dunkirk, Hamburg and Berlin, where they are crushed, and the oil passes into general commerce without maintaining its identity. Thus statistics concerning it are meagre.

Niger, Kersanee, or Ram-til Oil.—The "Niger seed" of African commerce, and the *ram-til* or *kersanee* of Indian cultivators, is the product of *Guizotia oleifera*. The plant grows wild on the Gold Coast of Africa, and is cultivated in Abyssinia, and in many parts of India, especially Mysore and the Deccan; here the seed is sown in July–August, after the first heavy rains, the fields being simply ploughed, and neither weeded nor manured. The crop is cut 3 months after the sowing, and, after being sun-dried for a few days, the seed is threshed out, the produce being about 2 bush. an acre. By the common country mills, only 25 per cent. of oil is got from the seed, but better appliances bring the average up to 35. The oil is limpid, clear, pale, and sweet-flavoured, and is used as an edible oil by the poorer classes of India, and commonly as a lamp-oil. Though much inferior to gingelly oil, it is frequently used as a substitute for it, and to adulterate both this and castor oil. The oil contains but little stearic or palmitic acid, hence soap made from it, though very white, is soft. The cake is an esteemed food for milch cows.

Dika Fat.—The seeds of *Irvingia Barteri*, the *dika* of West Africa, afford 60 per cent. of a solid fat resembling cacao butter, fusing at 86°–91½° F. (30°–33° C.), containing myristin and laurin, and capable of making very fine soaps. Much of the dika fat imported into this country is almost as hard as stearin, with a reddish colour from suspended impurities, and a fatty acid melting at above 120° F. (49° C.); its soap is very hard.

Shea, Galam, or Bambouk Butter. The c[illegible]e é, *shea*, or butter-tree of West Africa (*Butyrospermum* [*Bassi*[illegible]d a] *Parkii*) forms miles of forest on the south bank of th[illegible]e Niger, and the same or an allied species, locally termed *lul*[illegible]or *u*, is common on the river Djour, in the Bahr el Ghazal pr[illegible]llo-

vince, where its "butter" is used by the natives in cooking. The seeds, as large as pigeons' eggs, are exposed for several days to dry in the sun, and reduced to flour in a mortar; the flour is placed in a vessel, sprinkled with warm water, and kneaded to the consistence of dough. When the kneading has proceeded so far that greasy particles are detached by the addition of hot water, this last operation is repeated until the fat is completely separated, and rises to the surface. The fat is then collected and boiled over a strong fire, with constant skimming, to remove any remaining pulp. When sufficiently boiled, it is poured into a damp mould, and, when set, is wrapped in leaves, and will keep thus for 2 years. The yield is 30–40 per cent. The "butter" is greyish white, with sometimes a reddish tinge, and may be rendered quite white by repeated filtration in a warm closet; it resembles tallow in appearance, but is more unctuous, and greases the fingers; it has a faint characteristic odour, and a sweetish flavour. According to one authority, the fatty acid is margaric, and its melting-point is 142° F. (61° C.); according to others, there are 2 fatty acids, stearic and oleic, the melting-point of the mixture being 156° F. (69° C.). Yet another authority states the proportions as 7 of stearin to 3 of olein, and says that when acidified and distilled, it gives a yellow, crystalline fatty acid melting at 133° F. (56° C.), and when pressed, at 151° F. (66° C.), but that it cannot be used for candle-making, as it is comparatively soft, despite its high melting point, and is very liable to give a red smoky flame. It dissolves entirely in the cold in turpentine spirit, incompletely in ether, and is almost insoluble in alcohol. It saponifies readily with alkalies. There is also present in it, in the proportion of $\frac{1}{2}$–$\frac{3}{4}$ per cent., a substance resembling gutta-percha, and which has been called "gutta-shea." This is insoluble in alcohol, alcohol and ether mixed, acids and alkalies; but slightly soluble in pure ether, and in ordinary animal and vegetable fats. It can be removed from the fat by dissolving in a mixture of 3 parts ether and 1 alcohol, when it separates in a filmy state, more readily if the fat be

first saponified. It exists ready formed in the oil, some in suspension, removable by filtration, some in solution in the fat. The fat itself, shea butter, is imported into England to the extent of upwards of 500 tons annually, from Sierra Leone, for use in the manufacture of hard soaps, chiefly in combination with other oils. It is largely consumed in some of the Continental candle-factories. The natives employ it for food, for anointing and for lighting.

Vegetable Tallow is a sort of generic name for a number of hard fats obtained from various members of the vegetable kingdom in tropical and sub-tropical countries; the history of many of these products is often very hard to trace, and it not unfrequently happens that two such substances, nominally from the same district, vary considerably in value for soapmaking purposes. Hence the manufacturer will do well to test the merits of each lot, before purchasing it.

(*a*) *Chinese.*—The vegetable tallow of China is produced by the "tallow-tree" (*Stillingia* [*Croton, Sapium, Excœcaria*] *sebifera*). It is a native of China and the adjacent islands, and has been introduced and naturalised in India and the warmer parts of America. In China it is chiefly cultivated in the province of Chekiang and the adjacent Chusan Archipelago, in Kiangse and in Hoopih. In India it thrives in the North-West Provinces and the Punjab, especially at Paonee, in Gurhwal; at Ayar Tali and Hawul Baugh, in Kumaon; and in the Kangra Valley. The tree flourishes equally well on low alluvial plains, in the rich mould of canals, in sandy soils, and on mountain slopes. Its fruits are about ½ in. diameter, and contain 3 seeds thickly coated with a fatty substance, whence the "tallow" is obtained by treatment with steam and hot water. A very inferior oil is obtained from the seeds or kernels also, which are crushed between mill stones and steamed. The tallow itself is purified by melting and subsidence, and is then poured into moulding tubs, sprinkled inside with dried red earth to prevent adhesion. When cold, it is turned out in masses of 80 lb., a

hard, brittle, pure opaque white, tasteless and odourless fat. Its melting-point varies from 98½° to 111¼° F. (37°–44° C.), and its composition is almost pure stearin. The yield is about 20–30 per cent. of the weight of the seeds. Its chief and almost only application in China is for making candles, which are usually coated with wax; in India it has been tried as a lubricator; and it is found to burn well, without smoke or smell. An immense native trade in it is carried on in China.

(*b*) *Malayan.*—In Borneo, Java and Sumatra, are several species of *Hopea*, producing nuts which, when compressed, yield fatty oils, extensively used under the names of "vegetable tallow" and "vegetable wax." Three species of this genus are common in Sarawak; the one most valued for producing oil is a fine tree growing on the banks of the Sarawak river to a height of 40 ft; its fruits are produced in the greatest profusion about December–January, and are as large as walnuts. These nuts are collected by the natives, and yield a very large proportion of oil, which, on being allowed to cool, takes the consistence of sperm, and in appearance very much resembles that substance. In England, it has proved to be an excellent lubricator for steam machinery, far surpassing even olive oil; and it has been used in Manilla in the manufacture of candles, and found to answer admirably. As it becomes more common, it will doubtless be applied to many other purposes. This oil has nearly the consistence, and something of the appearance, of tallow, but is generally yellower. It is found in the markets in rolls 1½–3 in. in diameter. It is used in the interior almost exclusively for lighting and culinary purposes. A vegetable tallow is also afforded by the seeds of *Tetranthera laurifolia*, widely dispersed over Tropical Asia, and the Eastern Archipelago, as far south as New Guinea. In Java and Cochin China it is commonly used for making candles, notwithstanding its disagreeable odour.

Both Chinese and Malayan kinds of vegetable tallow, like shea butter, are glycerides, and contain about 95 per cent. of

saponifiable matter, which has much less olein in it than animal tallow.

(*c*) *African.*—A so-called vegetable tallow or butter is obtained in Sierra Leone from *Pentadesma butyracea;* the tree yields from its several parts, especially the fruit when cut, a yellow fatty juice. The fat of a species of *Pentadesma*, under the name of *kanya*, is used for culinary purposes in the neighbourhood of Zanzibar, and is said to remain sweet for a long time.

Corn Oil.—The oil contained in maize or Indian corn is used in the United States for soapmaking. In the process adopted in glucose works for the production of this oil, the corn, to begin with, is steeped in water, whereby the hull is loosened from the grains and becomes quite tough. Next, the corn is crushed between rollers. Examining such a crushed grain closely, it is found that the starch has been crumbled by the pressure, while the softened germ has remained whole, and forms one piece about the size of a grain of rice; the outer hull at the same time has merely burst into a few large pieces. When the crushed corn is next run into a tank partly filled with water, the starch and the particles of bran will sink to the bottom, while the germs will float on the surface. Here then the latter are obtained in the most favourable condition possible for extracting the oil by pressure. The qualities of the oil from the soapmaker's standpoint do not yet seem to be completely studied.

SECTION C.—ROSIN.

Rosin or **Colophony.**—The several kinds of rosin, colophony, &c., are the solid residues left after the distillation of the turpentines, which are themselves liquid oleo-resins obtained by tapping the trunks and branches of the *Coniferæ*, or pines. The so-called "pine barrens" of the United States extend from Virginia to the Gulf of Mexico, but the extraction of turpentine is an industry mainly developed

in North Carolina. American rosin is characterised by a peculiarly fragrant smell, in which French rosin is deficient, and this smell is retained by the soap made from the rosin. During the American civil war, when no rosin was to be had from thence, the small stores of it in England were so much valued for this fragrance in soap that soapmakers were compelled to pay more for American rosin than for tallow. The chief seat of the French rosin industry is in the departments of Landes and Gironde, where the extraction of the crude oleo-resin and its subsequent treatment are carried out in a more systematic manner than in America.

The rosin of commerce varies very much in colour; the finest grade is known as "window-glass," and all shades occur from this down to a black opaque rosin.

The lighter shades only can be used for better-class soaps. Such are liable to contain a little turpentine, which, to some slight extent, injuriously affects the hardness of the soap; and they are apt to become unpleasantly soft in very hot weather. Opaque rosins contain turpentine, and in rare cases, water. Dark rosins may be improved in colour, and deprived of suspended impurity by being melted, allowed to settle, and then boiled on a weak solution of common salt. Dark rosin may also be distilled with steam under a pressure of 10 atmos. to make it nearly white for soapmaking. Like many other distilled products, however, it has a tendency, both alone and in combination, to oxidise rapidly and deteriorate in colour. When fine rosin is unusually dear, however, this process may be employed with advantage. It is somewhat remarkable that if 2 rosins, French and American, of equally fine colour, be converted into soap, the soap from the American rosin will be some shades lighter than that from the French. In carelessly prepared rosin, bits of stick, leaves, &c. are apt to occur, all which, if they find their way into the soap-copper, seriously damage the colour of the ultimate product.

Rosin is never employed alone for soap, but always in conjunction with fats; it has been described as an ameliorator,

and is also a cheapener; it contributes to the popular qualities of soap, rendering it more readily soluble, and forming a copious lather in laundry and household soaps. In them the proportion varies from 15–20 per cent. of the fatty matter employed, to an equal weight, or even more. Hard soaps for manufacturing purposes rarely contain it. It may be saponified alone, and the result mixed with a fat-soap in due proportion, or the whole may be saponified together. The result is the same, and the choice of methods depends upon convenience in working; but the former is preferable with impure rosin, in order to give it as many changes of lye as possible, and thus to wash out suspended impurities, such as leaves, bits of stick, &c., alluded to above. Our annual imports of rosin average over 50,000 tons.

SECTION D.—SUNDRIES.

Saponifiable Waste Matters.—Besides the raw materials used by the soap-boiler, enumerated above, are various bye-products of other manufactures, among which may be mentioned—the olein or oleic acid from candle-works; the grease recovered from the wash-water of woollen factories, which is very apt to contain unsaponifiable oils, and should be employed with caution;* the "foots" from various oil-refineries, which either contain strong mineral acids, or are partly formed soaps when an alkaline refining process has been used; waste fats recovered by carbon bisulphide or petroleum spirit, also liable to contain unsaponifiable oils; "suint," or the grease derived from natural wool, accompanied by potash salts; and, especially in France, the yolks of eggs (of which the whites have been used in the preparation of albumen), which contain a considerable percentage of fatty

* As this grease can scarcely be used by the soapmaker without being previously distilled, the account of its recovery and preparation is deferred to Chap. XIV., upon the distillation of fatty matters, under the heading of Candles.

matter. This list might be largely increased, since the soap-pan appears to be the natural destination of any rough fat-containing material which can be turned to no other purpose.

The value of a soapmaking material is best ascertained by saponifying a weighed quantity (say 5–10 *grm.*) with soda, dissolving the soap in water, decomposing it with a mineral acid, and then washing, drying, weighing, and examining the resultant fatty acids, as will be described later on.

Water.—The purity of the water employed in the factory is a matter of great moment to the soap-boiler. As a rule, spring water should be avoided, and river, lake, or even rain water should be employed whenever possible. If it contains suspended impurities, these should invariably be removed by subsidence or filtration. Organic impurities, if colourless, may be disregarded. The great enemies of the soapmaker are the soluble salts of lime, of alkaline earths, and sometimes even of metals, occurring in natural waters, because all these bases form *insoluble soaps* in the soap-copper, and use up large quantities of fat to no purpose, since these insoluble soaps are of no market value themselves, and if disseminated in a marketable soap, injure its appearance greatly. The hardness of water may be determined by "Clark's test" (described in most text-books of chemistry), in which an alcoholic solution of pure soap is employed. The amount of soap wasted by hard waters may be ascertained from this table, in which the hardness is supposed to be caused simply by lime salts.

Degree of Hardness.	In 1000 lb. Water.		Soap decomposed by 1000 lb. Water.
	Lime.	Gypsum.	
	lb.	lb.	lb.
5°	0·25	0·61	3·66
10	0·50	1·23	7·31
15	0·76	1·84	11·12
20	1 01	2·45	14·75

If no other than hard water is available, one of the various plans for softening it should be adopted under the guidance of a chemist, such as the addition to the water of milk of lime, either alone or with barium chloride, or of sodium silicate.

Salt.—As will be seen in the sequel, common salt plays a very important part in soap-boiling, and what has been said with regard to water applies equally to it. The purest kind is rocksalt freed from insoluble matter; next in order comes salt from brine-springs, such as those in Worcestershire and Cheshire; while sea salt contains so many other salts besides sodium chloride, especially magnesium chloride, that it should be avoided if possible. When that is not the case, it should be dissolved in water, and the magnesia removed by the addition of sodium silicate, when the insoluble magnesium silicate should be allowed to subside before the brine is used. In connection with salt, it will be found convenient to remember that strong brine, or a saturated solution of salt, contains $\frac{1}{4}$ its weight or 25 per cent. of sodium chloride, that its sp. gr. is 42° Tw. (25° B.), and that if nothing else be present in solution, every degree Baumé of sp. gr. corresponds to 1 per cent. of salt.

CHAPTER IV.

RAW MATERIALS: Refining, Clarifying, and Bleaching.

The various processes under this head may at first be broadly divided into "mechanical" and "chemical," although each, especially the latter, is capable of many subdivisions. They have for their object, firstly, the removal of all extraneous matters from the oil, such as animal or vegetable fibre and tissue incidental to the modes of preparation (as in olive and other seed oils, badly-rendered tallow, &c.); secondly, the removal of resinous substances dissolved in the oil, of which the refining of cotton-seed oil is a notable example; thirdly, the removal of fraudulent admixtures, such as lime, glue, &c.; fourthly, the correction of rancidity; and fifthly, where the preceding operations do not sufficiently improve the colour of the oil, its bleaching by chemical processes. These will now be considered under their various heads, and it is obvious that much care and judgment are required in the selection of the particular method or combination of methods suitable to the refining of any given oil. The term oil will, in this connection, include melted fats, as well as those which are liquid at the ordinary air-temperature; almost all the operations described in this chapter can only be performed upon liquid substances.

The first and most important method, to be employed either alone or as a sequel to others, is that of simple but prolonged subsidence, on a large scale. Where necessary, the tanks employed may be heated by steam-coils or steam-jackets. These must be used with caution, however, since

convection currents are set up, which interfere materially with the steady deposition of impurities. The tanks should be provided with stopcocks or valves at various levels, in order to draw off separately the purified oil, and the "foots," which is a generic name for that concentration of impurities in a small quantity of oil, which accumulates at the bottom of vessels in which oil-refining is carried on. For example, olive oil is mostly subjected to no process of purification, beyond what is attained by allowing it to deposit impurities, and repeated decantation. But for the best qualities, further purification is necessary, not only to secure limpidity, but a capacity for lengthened preservation, by eliminating the water, mucilage, and parenchymatous matters, some of which, if not removed, are liable to act as ferments. Cocoanut oil is another example of purification by simple subsidence and filtration.

When prolonged subsidence fails to remove suspended impurities, filtration may be tried with advantage. In France the oil to be purified is received into perforated boxes carpeted with carded cotton (wadding); elsewhere, cotton tissue interposed between beds of granular and washed animal charcoal form the filter; also a bed of dry moss, on the "Grouvelle et Jaunez" system; also layers of sand, gypsum and coke; also alternate beds of sand and vegetable charcoal, according to Denis de Montfort's plan; also carbonised schist and peat, by Cossus' method; also clay heated to 200° F. (93° C.), as proposed by Wright; also by introducing China clay and allowing the oil to stand at a moderate temperature, then filtering through cotton, as adopted by A. Bizzarri. It is usual to assist the action of the filter in some way, mechanically, owing to the viscidity of oils. A simple mode of doing this is so to arrange the filter that there is a considerable pressure upon the filters from a "head" or column of the oil to be filtered. This may be conveniently carried out by using closed bags of filter-cloth confined in cylindrical wire cages as the filtering medium; the mouth of each bag is tied tightly over a stop-

cock connected with a main pipe which delivers the oil from a tank, and this tank may be placed at any desired level above the filters. With oils that are very thick, or even solid, at the atmospheric temperature, it is necessary to enclose the whole arrangement in a heated chamber. Another mode of hastening filtration is by utilising the atmospheric pressure upon the surface of the filter-bed; this is conveniently done by placing the filtering medium in any suitable vessel, to the bottom of which is adapted a pipe for the exit of the filtered oil, which pipe is connected with an air-pump and small engine, by which a partial vacuum is maintained in it. In this case the oil is practically sucked

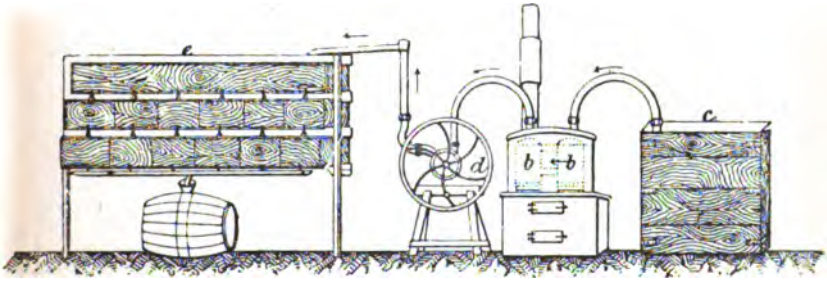


FIG. 18.—FILTERING OIL OUT OF CONTACT WITH AIR.

through the filter. Still another mode is that of Pietro Isnardi, of Livornia, Tuscany, which received an award at the Vienna Exhibition. This apparatus, Fig. 18, consists of a boiler full of water, serving as a water-bath for 2 turned-iron cylinders *b*, receiving the oil from the reservoir *c*, a suction and force-pump *d*, and a filter *e*, containing perforated trays whose holes are filled with wadding. This apparatus enables the oil to be filtered without coming into contact with the air, and at an elevated temperature which can be regularly maintained. As an adjunct to filters of any kind, some form of filter-press will be found very useful for removing the last traces of oil from the residues left on the filters.

When impurities are not removed by subsidence or filtration, recourse may be had to the action of acids or alkalies upon them. There are several methods of applying mineral acids to the purification of oils. Thénard's process consists in gradually adding 1–2 per cent. of strong sulphuric acid to oil previously heated to 100° F. (38° C.), and mixing by constant agitation.* When the action of the acid is complete, the oil, after 24 hours' rest, appears as a clear liquid, holding flocculent matter in suspension; a quantity of water heated to 140° F. (60° C.), equal to about ⅔ of the oil, is added, and the mixture is well agitated until it acquires a milky appearance. It is then allowed to settle for a few days, when the clarified oil rises to the surface, while the flocculent matter falls to the bottom with the acid liquid. The oil is then drawn off, and washed in another vessel by agitation with half its bulk of warm water; but it requires to be filtered to make it perfectly clear. This process is largely used for refining linseed oil. A mechanical mixer may be used, or the whole mass may be well agitated by the injection of finely divided streams of air, through a perforated pipe.

When fats have been treated with zinc (see under Glycerin) to effect separation of the glycerin, they become contaminated with the metal, and would discolour soap made from them. To prevent this, Bang and De Castro treat them while still hot with sulphurous acid in a lead-lined vessel.

Cogan operates upon about 100 gal. of oil with about 10 lb. of sulphuric acid, previously diluted with an equal bulk of water. This mixture is added to the oil in 3 portions, the oil being well stirred for about an hour after each addition. It is then stirred for 2–3 hours to ensure perfect mixture. After being allowed to stand for 12 hours, it is

* The first effect of the acid is a powerful dehydrating action, which removes the water that holds the impurities in solution; next it chars the impurities themselves, by which they are rendered insoluble and are partially destroyed.

transferred to a copper boiler with a perforated bottom, through which steam enters and passes in a finely divided state through the oil, raising it to the temperature of 212° F. (100° C.). This is continued for 6–7 hours, and the oil is transferred to a cooler, shaped like an inverted cone, terminating in a short pipe, and provided with a stopcock at the side, a little distance from the bottom. After standing till the liquids are separated, generally about 12 hours, the acid liquid is drawn off through the pipe at the bottom, and the clear oil by the stopcock in the side of the cooler; all below this tap is generally turbid, and is clarified by subsidence, or mixed with the next portion of oil.

These acid processes are efficient when well conducted, but too much or too little acid may spoil the product, because, as most of them depend for their action upon the fact that strong sulphuric acid chars organic substances by the removal from them of the elements of water, it chars the fibre in the oil first, but if more acid than necessary for this be present, it attacks the oil itself, and oil thus stained by charring cannot be completely decolorised again.

On this account perhaps, more general preference seems to be accorded to alkaline processes. Evrard's, which is chiefly applied to colza and rape oils, is as follows. The oil, drawn cold, or at very slight heat, is well crutched up with a weak lye of soda or potash, and allowed to settle. Two layers soon form—a neutral oil floating on an alkaline liquid, a mixed emulsion intervening. The alkaline liquid is drawn off, and replaced by slightly alkaline water, and the whole is left to settle. This is repeated a few times with clear water, till the liquid at the bottom of the settlers is only slightly milky. The oil is drawn off and filtered, and is superior to oil purified by sulphuric acid, being much less corrosive to metal. The turbid residual waters are treated with acid, and give a greasy product fit for soapmaking. A much simpler alkaline method adopted in Italy for olive oil is to add 400 *grm.* of ammonia, diluted with 800 *grm.* of water, to every 100 *kilo.* of oil; the whole is then thoroughly

agitated, and allowed to stand for three days, after which the clear oil is decanted and filtered.

Hopkinson makes an insoluble lime soap of the impure grease, and then dissolves out the fat by a volatile solvent, separating afterwards by distillation.

Winter and Colman heat the fatty matter to liquefaction and treat it with 1–15 per cent. of powdered fuller's earth well agitated together. The purified fat floats above the impurities. In this connection it is interesting to notice the large quantities of fuller's earth which are sent from this country to the United States, notably Chicago, for purifying lard.

Tichenor applies an electric current for purifying fats.

Nordlinger prepares a "purifying oil" by dissolving in it a metallic soap (cadmium, copper, iron, lead, manganese, or zinc salts of the higher fatty acids) at a temperature of about 302° F. (150° C.), and clarifying by subsidence. A small quantity (5–10 per cent.) of this "purifying oil" is added to impure vegetable oils, and, on standing for some time, the mucilaginous matters are precipitated by the chemical reactions set up, and the clear supernatant oil can be drawn off.

For linseed oil, Hartley and Blenkinsop use manganese linoleate dissolved in oil of turpentine, about $\frac{1}{8}$ part per cent. sufficing; but if much mucilage is present, a preliminary treatment with sulphuric acid is given.

One of the most remarkable impurities in fats, arising from methods of preparation merely, is that of lime in bone fat. This fat has the power of dissolving considerable quantities of lime salts, especially phosphate and carbonate. No amount of subsidence or filtration will remove them, and their presence in a soap-copper is most objectionable. It is greatly to be desired, therefore, that English makers of this fat would follow the example of their American confrères, and boil their bone grease, after removal from the extractors, with a weak solution of sulphuric acid, in lead-lined wooden tanks. This removes all the lime in the form of sulphate

which deposits on the floor of the tank after due subsidence; it also removes the gelatin and extraneous water entangled in the bone fat, which cause the crude grease to froth greatly when heated.

A good example of the removal of resinous substances from oils is afforded by the process adopted for refining and bleaching cotton-seed oil, an industry which has enormously developed, both in England and the United States, within the last 25 years. When freshly expressed from new seed, this oil is of a light-claret colour, which darkens by long keeping, in which case also the oil becomes more viscid, probably from oxidation of some of its constituents. The colouring matter is almost entirely rosin, which may be removed by agitation at about 140° F. (60° C.), with a solution of sodium carbonate. It is found in practice, however, that a much better result is obtained by the use of a caustic alkali—solution of soda or potash, or in some rare cases, milk of lime. The amount of alkali thus employed depends entirely upon the quality of the crude oil, and is best determined by a preliminary experiment upon a small scale. A solution of caustic soda at about 20° Tw. (13° B.) is a suitable strength. Agitation must be thorough, and may be effected by any convenient mechanical means, or by the injection of air, before alluded to. The process is a rapid one; if the saponaceous liquid does not readily separate from the oil, the addition of a little brine will cause it to do so. The operation is often divided into 2 or 3 stages, and occasionally the refined oil is further bleached by one of the oxidation processes, such as by chloride of lime or "bleach."

After all the refining, it should be washed with warm water, allowed to settle and decanted, or filtered at as low a temperature as possible, especially if an oil be desired that will remain fluid at a low temperature. This process will answer well for any oil containing rosin. The imperfect soap, after removal, is treated with enough mineral acid to remove all the soda, and the resulting mixture of rosin, fatty

acids, and neutral oil is distilled with superheated steam (see Chap. XIV.) for the manufacture of fatty acids, the rosin being left in the still as pitch. The chief seat of this industry in England is at Hull. In the United States, the quantity of rosin is so small, that the "foots" from the cotton-seed oil refineries are made into a curd soap.

For the removal of fraudulent admixtures from commercial oils, no general rule can be given; but subsidence, filtration, and boiling with weak sulphuric acid, will generally effect the desired result. Special modes are best sought under the head of Proximate Analysis, which forms the subject of the next chapter. Some of the methods there recommended for the laboratory, are capable of being carried out on a large scale.

Methods for correcting rancidity in oil are as follows:—(*a*) Agitation with 5 parts of good vinegar, repeating the operation several times. (*b*) Agitation (5–6 times) of 50 parts of oil with 80 parts of water at 86° F. (30° C.) holding 12 parts of common salt in solution. (*c*) To 100 *litres* of oil, are added 2 *kilo.* of calcined magnesia; the mixture is agitated 4 times daily for ¼-hour each time for 6 days; the oil is then filtered; it must be quickly used, or it will become rancid again. (*d*) Agitation with a weak solution of caustic alkali, or with a moderately strong one of an alkaline carbonate. (*e*) Prolonged agitation with water.

Most processes for refining and bleaching oils also deodorise them to a certain extent. As many of the odorant principles are more volatile than the oils, they may occasionally be removed by merely heating the oil in a closed vessel provided with an exit-pipe. For destroying the disagreeable smell of coconut oil for soap-making, it has been recommended to boil it in a wooden vessel by free steam on water containing 6 lb. sulphuric and 12 lb. hydrochloric acid to each ton of oil. Prolonged steaming will sometimes remove the unpleasant odour characteristic of oily distilled products, although it is liable to damage their colour.

Many plans of decolorising oils are in vogue:—(*a*) Exposure to sunlight in large white glass bottles: the oil soon becomes colourless, but acquires an almost rancid flavour. (*b*) Agitation with 2 per cent. of a solution of potassium permanganate: bleaches effectually, but also leaves a bad flavour. (*c*) The oil is first agitated with water containing gum, and to the emulsion thus formed, is added coarsely crushed wood-charcoal; the whole is then slowly warmed to a degree not reaching 212° F. (100° C.), and when cold, the oil is dissolved out by ether or petroleum spirit, and the latter is recovered by distillation: the result is good. (*d*) A process often recommended is to pass nitrous acid gas through the oil.* (*e*) The oil (500 parts) is clarified by addition of 50 parts of China clay and 50 of water. (*f*) In some cases it is found advisable to use the coagulation of albumen in clarifying oils. The oil to be treated is mixed by agitation at the ordinary air-temperature with a weak solution of albumen in water. The whole is then gradually heated, most conveniently by steam, and when hot enough to coagulate the albumen, this latter collects in clots, enclosing particles of impurity; after the lapse of sufficient time, these clots subside, and the clarified oil is removed by decantation. The process is analogous to that of refining sugar syrups by the serum of blood.

Many oils are partially or completely decolorised by filtration through, or agitation with, freshly-burnt animal charcoal or boneblack. The apparatus for filtering is similar to that employed in sugar refineries, and consists essentially of tall wrought-iron cylinders filled with boneblack, and provided with a steam-jacket to control their temperature. When the charcoal ceases to decolorise, it should be treated with some solvent (carbon bisulphide, or petroleum spirit) to remove the oil, before it is revivified by calcination.

Most processes for the bleaching of oils depend upon the oxidation of the colouring matter by some suitable reagent,

* See subsequent paragraph, pp. [redacted].

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chiefly evolving nascent oxygen in some form. There are, however, instances known in which the colour is destroyed by a reducing agent, such as sulphurous acid, in an aqueous solution, as gas, or arising from the decomposition of an alkaline hyposulphite (e. g. that of sodium) by a strong mineral acid. It may be laid down as a general rule that oils which have been burned or charred by any previous process cannot be satisfactorily bleached. Experiment alone can determine the particular process best suited to any given oil, having regard to the purpose for which it is to be used. The utmost care is required in using any oxidation process for oils intended to be converted into soap, since if the oil itself be oxidised in any perceptible degree, as well as the colouring matter (i. e. if too much of the bleaching reagent be used), the resulting soap will often be worse in colour than if the oil had not been bleached at all, owing to the rapid formation of the same complex acids which gradually make their appearance in rancid tallow.

Palm oil and tallow are the two chief fats bleached by the soapmaker. Both may be bleached by pumping air into them in finely divided streams, while they are kept at about 180°–200° F. (82°–93° C.). The colour of tallow may also be removed by boiling it in lead-lined tanks upon a solution of chloride of lime, or of potassium chlorate, to which a strong mineral acid has been added. No more potassium chlorate than 0·3 per cent. on the tallow should be employed. Another mode of bleaching tallow, especially for dip-candle making, is to agitate it with the clear solution prepared by digesting manganese black oxide with dilute sulphuric acid, and then allowing the mixture to settle.

Linseed oil is bleached by Hartley and Blenkinsop by blowing a current of air through oil to which about ·125 per cent. of manganese linoleate has been added, at a temperature of about 190° F. (88° C.), the manganese salt acting as a carrier of oxygen.

Teal exposes linseed or rape oil in a finely divided state

to the influence of air heated to about 170° F. (71° C.), instead of heating the oil and dividing the air.

Mills applies a mixture of hot air and volatilised sulphurous acid, under a pressure of two atmospheres.

Hydrogen peroxide is too costly for general use.

Experiment has shown that the colour of palm oil may be quite destroyed by heat. To effect this, the oil may be kept for some hours at about 260° F. (127° C.), or it may be put into a closed, horizontal, iron cylinder, and rapidly heated by a fire beneath up to about 464° F. (240° C.), at which temperature the colour is destroyed. This process gives rise to most offensive vapours, especially acrolein, and necessitates the conduct of operations in a closed vessel, with suitable means of condensing the vapours and rendering them innocuous; as they come off at a high temperature, they must not be conducted into a flue until they have been cooled down, and impregnated with water. The details of such an apparatus, which may be applied to other operations, will be further described in a later chapter, dealing with legislative enactments affecting the soap trade.

Palm oil may also be very suitably bleached by potassium bichromate and hydrochloric acid. The oil is made as free as possible from impurities, and, at about 120°–130° F. (49°–54° C.), is agitated with a strong solution of potassium bichromate, containing about 1 lb. of the salt to every 100 lb. of oil. To this is added enough hydrochloric acid to form chromium sesquichloride with all the chromium in the bichromate, the quantity of liquid acid necessary of course varying with the amount of real acid contained in it. A slight excess of acid is rather an advantage than otherwise. The process occupies about an hour, after which, subsidence removes most of the chemicals, while subsequent agitation with hot water renders the oil quite pure enough for the soap-copper, although it is a curious fact that palm oil has a strong solvent action upon chromium salts, so that it is impossible to remove all the green colour from the oil by washing it, and hence a considerable precipitation of

hydrated chromium oxide takes place in the soap-copper. This should be carefully removed with the spent lye, else the result in the soap will be most unsightly. Recently, sodium bichromate has been offered as a commercial product, at a less price than the corresponding potassium salt.

Several years ago, when palm oil was much more used in soapmaking, and "bichrome" was much dearer, than at present, the chromium oxide was thus recovered, and used over again. Excess of milk of lime was added to the solution, and the resulting mixture of lime and chromium oxide was dried by a gentle heat, and finally roasted at a comparatively low temperature, till it assumed a lively yellow colour. This rough calcium chromate was then digested in the cold with a slight excess of hydrochloric acid, so that the solution contained calcium chloride and impure chromic acid, and was used for bleaching fresh quantities of oil. In this way the same chromium did duty many times over, but, for reasons given above, there was always a certain loss of chromium in the series of operations. By whatever process palm oil is bleached, it should be saponified as soon as possible after bleaching, since it is apt to deteriorate in colour (especially if it is allowed to get cold, and is then melted again), becoming more or less brown; the same remark applies, to a less extent, to other bleached fats.

A very effective mode of decolorising fatty matters is to distil them in an atmosphere of superheated steam. This process will be described in Chap. XIV., but it may be useful here to warn the inexperienced soapmaker, that fatty matters which have been distilled are apt to deteriorate in colour (instead of improving, like a neutral fat), under the influence of air and light, also every time that they are melted and cooled again. Moreover, the colour of a soap from distilled material is decidedly worse than the colour of that from a neutral fat of the same shade.

In some textbooks, and by many adventurers, soapmakers are advised to treat comparatively fluid oils with nitrous acid, in order (1) to bleach them and (2) to solidify them.

This is a process which it is as well to avoid; both the bleaching and the hardening effects are apparently very successful; the colour of the oil, however, rapidly resumes its original tint, or worse; and the soap made from the solidified oil is no harder, and often worse in colour, than that from the oil before such treatment.

There is, however, a very scientific process for the solidification of liquid oils, which has been worked out industrially by St. Cyr Radisson, of Marseilles. It depends on the laboratory reaction discovered by Warentrapp, that when oleic acid (or olein) is heated with an excess of caustic potash, it is resolved into palmitic acid, acetic acid, and hydrogen. Radisson has shown that caustic soda will effect the same change. Although the process has hitherto been confined to the conversion of oleic acid into palmitic acid, for candle-making, it is applicable to any fluid saponifiable oil. It requires, however, that the fatty acids so produced shall be distilled in order to be made sufficiently good in colour, and hence an account of it is reserved for Chap. XIV., in the part of this book relating to Candles.

Very complete machinery for refining cotton, linseed and rape oils, and workmen skilled in both mechanical and chemical methods of refining oils, are provided by Rose, Downs & Thompson, Hull.

One other mode of treatment of neutral fats, preliminary to their saponification, remains to be noticed, and is of recent origin. It is that by which these neutral fats are deprived of their glycerin before being saponified with soda (or potash). As will be seen in the sequel, the problem of recovering glycerin from the "spent lye" of the soapmaker, is a very difficult one, and in the opinion of several who are well qualified to judge, all glycerin in future will be made from the fats direct, before they are saponified, and not recovered from the spent lye. Detailed information on this subject will be found in Chap. XVI.

CHAPTER V.

RAW MATERIALS;—Their Proximate Analysis.

The object of this chapter is to deal with comparatively simple methods of testing the purity, or otherwise, of the various fatty matters employed by the soapmaker, and to point out the most probable impurities in and adulterations of them, so as to enable the manufacturer to form a true judgment as to the actual value to him of any saponifiable material. The necessary tests of the alkali employed will be explained in Chap. VI., which is devoted to the preparation of caustic alkali, while the ordinary analysis of water, and of the various mineral salts employed in soap manufacture, is of a kind which is best obtained by practical instruction in any laboratory, assisted by a textbook of inorganic analysis, of which there are very many published. A general acquaintance with laboratory manipulation will therefore be assumed.

The ordinary solid fats and fixed oils (with the exception of butter and a few others) may be looked upon as mixed glycerides of oleic, stearic and palmitic acids, in various proportions, the first preponderating in the oils, and the two last (especially stearin) in the fats. For ordinary purposes, there are therefore the following constituents to determine:—(1) Moisture, present in greater or less quantity in every commercial sample of fat: (2) organic suspended matter, such as impurities from animal and vegetable tissue; (3) mineral matters, such as lime in bone grease; (4) free fatty acids, arising mainly from the rancidity of fats; (5) total fatty acids, in any ordinary oil or fat; (6) oleic, stearic and

palmitic acids, in any ordinary oil or fat; (7) soluble and insoluble fatty acids, only necessary in butter, and the few exceptional fats similarly constituted (coconut and palm-nut oils contain some soluble fatty acids); (8) glycerin, from which to calculate the glyceryl in the fat; (9) possible presence of paraffin wax and mineral oils.

It is desirable to point out here the great importance of obtaining for analysis a sample which shall really represent the bulk fairly. In some cases, this is much more difficult than is generally supposed. Ordinary casks or barrels of tallow, from well-known brands, present little or no difficulty, provided that the borer is driven well into the middle of the cask. In the case of palm oil, the circumstances under which the casks are gradually filled, and kept in a semi-liquid state, render such a simple proceeding quite valueless, since different parts of the cask are very liable to contain quite different proportions of water and of "dreg," the generic name given to solid impurities in raw fats. Hence in sampling palm oil, it is necessary to employ a special kind of borer, provided with a slide, which shall take a complete section of the contents of the cask from one side to the other, and, if need be, from one end to the other also. These sections, weighing some ounces, must then be thoroughly incorporated. The same precautions should be applied in the case of greases of various kinds, which are often poured into their casks before they have deposited all their impurities in the tank, so that the process of subsidence is continued in the cask, producing layers of various composition. In the case of liquid oils, the casks should be well rolled about, in order to mix their contents thoroughly, just before sampling them. Rosins, as a rule, are very rarely adulterated, and their mode of manufacture is such, that a piece taken out of the head of a cask is usually a fair sample.

(1) *Estimation of Moisture in Fats.*—25 *grm.* of the fat are weighed into a carefully tared porcelain dish, or a small beaker, which is then placed over a low gas flame, and stirred with a thermometer, taking care that the temperature

is maintained considerably above 212° F. (100° C.), but not exceeding 275° F. (135° C.). This is continued until no more bubbles of vapour escape, indicating that all the moisture has been expelled; the thermometer is then carefully drawn out, and any fat remaining attached to it is scraped off and returned. If, however, an instrument with a long narrow bulb be used, and it be carefully turned round while it is drawn out, usually no fat will adhere on withdrawing it. The dish plus the fat is then weighed, and the tare being deducted, the remainder is the dry fat in the 25 *grm.* taken, which, multiplied by 4, gives percentage of pure fat, and the difference between that and 100 represents the percentage of moisture. Some fats, especially bone grease, skin grease, &c., will froth very much during the first part of this operation, so that the vessel employed should be a capacious one, and the temperature should not be raised too rapidly; until, however, 275° F. (135° C.) is reached, it is not safe to assume that all water is expelled. Cocoanut and palm-nut oils, thus treated, are apt to lose a small fraction of 1 per cent. of the oily constituents, but scarcely any other oil does so, unless the sample be maintained long at the highest point. Any mineral acids likely to be present are volatilised at the same time, except sulphuric acid, a notable quantity of which will discolour the oil at the above-named temperature.

Another method is to keep a weighed sample of the fat in an air-bath at 230° F. (110° C.) until it ceases to lose weight; but the time required for this operation is rather longer than for the method previously given, and hence the result is not obtained so quickly.

As an illustration of the great range in percentage of moisture, it may be stated that while good tallow should not exceed 0·25 per cent., certain kinds of palm oil and inferior butter exceed thirty per cent.

(2) and (3) *Estimation of Organic and Mineral matters present as Impurities in Fats.*—This applies to the estimation of fibrous and mineral impurities in oils and fats, of curd and salt in

butter, &c., and is thus conducted. The contents of the dish already used for moisture are melted, and the melted fat is poured off as far as possible without disturbing the sediment. Some petroleum spirit, rectified at a temperature not exceeding 188½° F. (87° C.), or some carbon bisulphide, is poured into the dish, and the whole is well stirred, and transferred to a previously weighed filter. By means of successive portions of the solvent, the whole of the contents of the dish are washed on to the filter, and all traces of fat are completely washed away from the other matters, which remain on the paper. The filter is then dried at 212° F. (100° C.), weighed, and the tare having been deducted, the remainder is organic matter plus mineral matter (or, in a butter, curd plus salt).

The filter and contents may then be transferred to a previously weighed platinum crucible, and heated for some time to dull redness, till the ash becomes greyish-white. The crucible and ash are weighed, and the weight of the former being deducted, the difference is mineral matter (or, in a butter, salt). The mineral matter thus found is deducted from the former result, and the difference is the organic matter; each being multiplied by 4 gives the respective percentages.

There are certain cases, however, in which impurities are actually dissolved in the fats, and hence are not estimated by this method. Bone grease is a good example of this, holding as it does, lime salts in solution. In such cases it is well to boil the fat upon dilute sulphuric acid, then upon water, and afterwards to proceed as above, weighing the pure fat ultimately obtained. Or a weighed quantity of the fat may be ignited, either alone or after being mixed with mercury oxide. In the case of palm oil, and similar imperfectly purified fats, it will generally be found that the percentage of "dry dreg" is from one-third to one-half that of the water; hence the mere estimation of the water (a comparatively rapid operation) affords good material for an approximately correct valuation of the sample.

(4) *Estimation of the Free Fatty Acids in Fats.*—This is a point to which attention has only recently been directed, but

as it is of more importance in relation to the use of fats and oils as lubricants, than for conversion into soaps or candles, it will only be shortly dealt with here.* About 5 *grm.* of the fat is taken (the absence of mineral acid being first determined) and dissolved in a suitable flask in 50 *c.c.* of strong rectified spirit of wine (about 0·82 sp. gr.), by boiling for 3 minutes in a water-bath, a vertical condenser being attached to the flask, which is shaken round once or twice while in the bath. The reagent employed is alcoholic potash, containing caustic potash equal to 28·05 *grm.* of pure potassium hydrate per litre, which is cautiously added from a burette. The indicator is an alcoholic solution of phenolphthaleïn, whose crimson colour is instantaneously discharged until neutrality is reached, when the pink colour produced by a slight excess of potash fades with sufficient slowness to leave, in most cases, no doubt as to the point at which to stop. From the amount of potash used, the acidity can readily be calculated. In the case of tallow, since the mean of the equivalent weights of stearic, palmitic, and oleic acids is 274, the potash acidity percentages multiplied by 5 will probably not be very far from the truth. This factor, however, must vary in other fats and oils, owing to the differences in the equivalent weights of their fatty acids.

(5) *Estimation of the total Fatty Acids in any ordinary Fat or Fixed Oil not containing Glycerides of Soluble Acids.*—This process, which is also applicable to the estimation of the total insoluble acids in a fat containing glycerides of soluble acids, divides itself into 2 heads, as follows:—

(*a*) Preparation of the sample.—If the sample be a perfectly clear and dry oil, it is at once ready for use; but if it be at all turbid, or if a solid fat, a portion must be placed in a tube, and kept in the water-oven below 212° F. (100° C.), until any moisture and heavy suspended impurities have

* A valuable résumé, and some original investigations of the subject, will be found in a paper by W. H. Deering, in the 'Journal of the Society of Chemical Industry,' iii. Also a good paper by L. Archbutt in the *Analyst*, vol. ix., may be consulted with advantage.

settled to the bottom. A well-dried filter-paper is then placed in a funnel over a dry beaker, also in the water-oven, and the nearly clear upper portion of the melted fat is filtered, until a sufficient quantity is thus obtained fit for analysis.

(β) Process of analysis.—A perfectly clean and dry 5-oz. flask is accurately tared on the balance, and 5 *grm.* of the melted fat are carefully weighed into it. (It is not important exactly to a fraction, but as nearly 5 *grm.* as possible should be taken, and, in any case, the weight must be noted with great care.) To this are then added about 30 *c.c.* of methylated spirit 60 o.p., and a fragment of caustic potash weighing about 2 *grm.*, and the flask is then placed in a basin of boiling water, until the whole of both fat and potash have dissolved, and the addition of a little water produces no permanent turbidity, which will be attained within 10 minutes, as a rule. The contents of the flask are then poured into a basin, and the flask is washed out with repeated quantities of boiling distilled water, until the contents of the basin measure about 250 *c.c.*, and no trace of soap remains in the flask. The basin is then placed over a low gas flame, and evaporated till it ceases to give off spirituous vapours, a little boiling distilled water being added, if necessary, to prevent too great a loss by evaporation. After simmering in this way for some time, the contents of the basin are made up to 300 or 400 *c.c.* with distilled water, and while gently boiling, a slight excess of hydrochloric or dilute sulphuric acid is added to them. The whole is then stirred and kept at a point just short of ebullition (to avoid loss by spirting) until a *perfectly clear* layer of fatty acids separates on the surface. If time be an object, the basin must now be cooled by immersion in cold water, and when the cake of fatty acids is solid, the watery liquid is poured off, and some distilled water is added, which in its turn is boiled, cooled and poured off. If the fatty acids are not sufficiently solid to make a good cake, a known weight (2 or 3 *grm.*) of stearic acid or beeswax may be added with advantage. After washing and draining, the fatty-acid cake may be carefully dried with filter-paper,

taking care that the latter absorbs no oil; it is then transferred to a weighed beaker or capsule, dried at a temperature of about 230° F. (110° C.), and weighed. If any fat remains adherent to the basin, the latter should be rinsed and dried; the fat can then be dissolved off by petroleum spirit or some other solvent, and its solution placed in another capsule, dried and weighed. From the total weight of fatty acids obtained, their percentage in the original fat can be readily calculated.

(6) *Estimation of Oleic Acid apart from the Palmitic and Stearic Acids.*—To do this, advantage is taken of the solubility of lead oleate in ether, so enabling its separation from the stearate or palmitate of the same metal. As formerly conducted, this was a tedious and not over accurate process; but by the apparatus and process devised by Dr. Muter, and here described, the estimation is rendered simple.

"A small quantity (not more than 1·5 *grm.*) of the purified fat is saponified by alcoholic potash in a flask, washed into a basin with boiling distilled water, and the alcohol is removed by evaporation, all as described before in the estimation of the total fatty acids. The solution is kept boiling, and treated with acetic acid, drop by drop, until a decided *permanent* turbidity is produced; dilute solution of caustic potash is then added by drops, with *constant stirring*, until the liquid *just clears again.* The clear solution is then precipitated by plumbic acetate in slight excess, and stirred until the precipitated soap settles thoroughly. The supernatant liquor is poured off, and the soap is at once washed by boiling with a large volume of distilled water, and decanting. By this process are obtained the perfectly neutral lead salts, containing:—Plumbic oleate ($Pb\ 2C_{18}H_{33}O_2$), plumbic palmitate ($Pb\ 2C_{16}H_{31}O_2$), and plumbic stearate ($Pb\ 2C_{18}H_{35}O_2$). The first is readily soluble in ether; the two last are quite insoluble. The soap is scraped from the basin with a platinum spatula, and transferred to a flask of 100 *c.c.* capacity. The basin is rinsed into the flask with absolute ether, and then the flask is filled up with the same solvent, corked, shaken

at intervals for some hours, and finally set to subside. The whole is then filtered through white blotting-paper, and the precipitate is washed with ether, till the washings cease to blacken with ammonium hydrosulphide. The filtrate and washings (which should not exceed 200 *c.c.*) contain the plumbic oleate, while the palmitate and stearate remain on the filter. Having thus got a solution of pure neutral oleate of lead in ether, it is transferred to a long tube of 250 *c.c.*, graduated from the bottom upwards, furnished with a well-ground stopper, and having a stopcock placed at 50 *c.c.* from the bottom (Fig. 19). About 20 *c.c.* of a mixture of 1 part hydrochloric acid and 2 parts water are then added; the tube is stoppered, well shaken, and set to subside, when a clear solution of oleic acid remains, the plumbic chloride sinking to the bottom. When sufficiently settled, a fixed portion of the ethereous solution is run off through the stopcock into a tared platinum dish, evaporated at a gentle heat, then dried at 212° F. (100° C.), and the oleic acid is weighed and calculated to the whole bulk. To make sure, it is well to run off two different quantities, and weigh them, so checking one by the other."

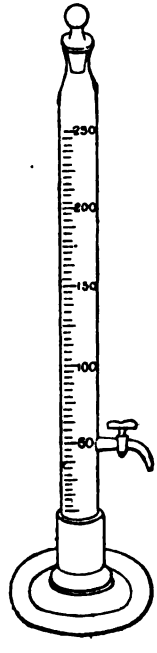


FIG. 19.
OLEIN TUBE.

In the case of drying oils, it is advisable to evaporate the ether, and to dry the fatty acids in a stream of hydrogen, rather than in the open air.

The residue left on the filter is transferred to a basin, heated with dilute acid, and treated as described in the last section, the result being a cake of stearic and palmitic acids.

7. *Analysis of Fats containing both soluble and insoluble Fatty Acids.*—To this class belong coconut and palm-nut oils, butter, and a few other fats. The process is performed

as follows:—5 *grm.* of melted and purified butter fat (as directed under "total fatty acids") are weighed into a 5-oz. flask, 50 *c.c.* of alcoholic potash containing 60 *grm.* ordinary caustic potash per *litre*), are carefully added from a burette, and the whole is boiled on a water-bath for about 15 minutes, or until the addition of a little water produces no turbidity. This solution is washed into a long, narrow, graduated measure with successive quantities of distilled water, till the whole measures 300 *c.c.*, and it is then divided into 2 parts of 150 *c.c.* each. In part A, the insoluble acids are estimated; in part B, the soluble.

Part A is treated with solution of barium chloride, until no more precipitate forms; the precipitate is collected on a filter, and well washed with warm water. It is then transferred to a "Muter's olein tube" (see Fig. 19) having a wide mouth, by washing it in with distilled water, and allowed to settle. As much as possible of the water is drawn off by inclining the tube forward and running off the clear water at the stopcock; 20 *c.c.* of diluted hydrochloric acid (1 acid to 2 water) are added, together with 100 *c.c.* of pure ether; the stopper is introduced, the tube is well shaken, and then allowed to settle till the ethereous solution separates. The amount of the ethereous solution is noted, and a definite quantity (say ½) is drawn off into a tared platinum capsule; the ether having been evaporated off, the residual acids are weighed.

Part B is diluted with another 100 *c.c.* of water, placed in a flask, and brought under a burette containing sulphuric acid of known strength, a slight excess of which is run in. The flask is then attached to an upright condenser, boiled until the insoluble acids separate in a clear oily layer, and then allowed to cool. The cake is detached, and the fluid is run off through a filter. Another 100 *c.c.* of boiling water is then added to the cake, and the whole is again boiled under the upright condenser; when cooled, the liquid is passed through the same filter. This operation is repeated, and the united filtrates are brought under a burette con-

taining a solution of sodium hydrate, of such strength that 1 *c.c.* of it corresponds to 1 *c.c.* of the acid, and, a few drops of alcoholic solution of phenol-phthaleïn having been added, the solution is run in. When a pink colour has been produced, the number of *c.c.* used is noted, and from this, the quantity of soluble fatty acids present can be readily calculated.

8. *Estimation of Glycerin in Fats.*—This is very rarely wanted except for scientific purposes, and it is a very difficult operation to perform satisfactorily. A comparatively simple process is that proposed by Dr. Muter, and described in detail in the *Analyst* for March 1881.

9. *Estimation of Unsaponifiable Oils in Fats.*—A convenient quantity, say 10 or 20 *grm.* of the fat is thoroughly saponified with an alcoholic solution of caustic soda; a few *grm.* of sodium bicarbonate are then added, and the whole is well stirred; 50 *grm.* of sand are then incorporated with the mixture, the evaporation being continued till a dry residue is obtained. This is then macerated with petroleum spirit, and the whole is thrown on a percolator and washed with more spirit, until a few drops leave no residue on evaporation. The whole quantity of spirit used is well mixed together, and from an aliquot part of it, the solvent is distilled off, and the residue is weighed. This process gives good results with comparatively pure oils, but when much mineral oil is present, portions of the dry soap dissolve with it in the petroleum spirit.

Another method has been worked out by A. H. Allen, which is capable of general application.* A solution is prepared by dissolving 80 *grm.* of caustic potash, or 60 *grm.* of caustic soda, in 1 *litre* of redistilled methylated spirit. To about 5 *grm.* of the fatty substance to be tested, 25 *c.c.* of this solution are added in a hemispherical porcelain dish,

* 'The Practice of Commercial Organic Analysis,' by Alfred H. Allen, 1882 edition, vol. ii. pp. 120, 121 and 165–169. This book may be consulted with great advantage by those concerned in the industry of oils and fats. See also *Chemical News*, vol. xliv. p. 161, and 'Journal of the Society of Chemical Industry,' vol. ii. pp. 49–58, 100–103 and 435–438.

about 5 in. in diameter; the whole is stirred, and gently boiled till all the alcohol is expelled, and the residual liquid froths strongly. If incomplete saponification be suspected, 10 *c.c.* more alcohol may be added, and the operation repeated. The contents of the basin are brought to a volume of 70–80 *c.c.* with warm water, and are then transferred to a pear-shaped separator furnished with a tap below and a stopper above, and there thoroughly agitated with about 50 *c.c.* of ether. After subsidence and separation, the aqueous solution is run off into a beaker, and the ethereous solution into a tared flask. The ether is distilled off; the residue, which may contain the higher alcohols from waxes, sperm oil, or bottle-nose oil, rosin oil, paraffin wax, or mineral oil, is dried at 212° F. (100° C.) and weighed.

TABLE.

Dissolved by the Ether.	*Aqueous Solution.*
Hydrocarbon oils.	
Neutral resins.	
Unsaponified fat.	
Unsaponifiable matter, as cholesterin.	
Myricyl alcohol (from beeswax).	
Cetyl alcohol (from spermaceti).	
Spermyl alcohol (from sperm oil).	
Colouring matters, as from palm oil.	
	Fatty acids, Resin acids, Carbolic acid, Cresylic acids — as potassium salts.
	Glycerin.
	Excess of caustic alkali.

SPECIFIC GRAVITY OF FATS.

It may be convenient here to discuss the subject of the specific gravity of fats, since there is often much to be learnt from a careful observation of this, especially when perfectly pure standards of undoubted genuineness can be obtained for the purpose of comparison. The differences, however, are so very slight, and the phenomena in the case of mixtures so remarkable, that great care is necessary both in the observation and in the inferences drawn from it. Moreover, there exists unfortunately great confusion in the mode of stating specific gravities. Thus:—the "specific gravity of butter is 0·912 at 100° F. (37·8° C.)," may mean that

the weight of a given volume of butter at 100° F. divided by the weight of the same volume of water at 60° F. (or even at 32° F.) gives a quotient of 0·912; or it may mean that the weight of the volume of butter at 100° F. divided by the weight of the same volume of water also at 100° F. gives a quotient of 0·912. This last is the true way of stating the case, and the term "actual density" was applied to it by Dr. Muter. There are several modes of making the observation; perhaps the most accurate is the employment of a "Sprengel's specific gravity tube," which is a U-tube whose legs are drawn out to a capillary bore, and bent at right angles, one being contracted to 0·25 *m.m.* diameter, the other, which has a mark near its end, to 0·5 *m.m.* This wider end is that which is inserted into the liquid during the process of filling, the other being attached by a piece of fine rubber tubing to an aspirator. By means of this apparatus, the specific gravity of any fat may be ascertained at 212° F. (100° C.), as follows: it is filled with water by exhausting the air from it, and supported in the neck of a 20-oz. flask (Fig. 20) in the bottom of which water is kept boiling briskly. The mouth of the flask should be closed by a watch-glass or porcelain crucible cover, so as to prevent cooling. When no further expansion of the contents of the tube is observed to occur, its nose is touched with a piece of filter-paper, and the tube is removed, wiped dry, cooled and weighed. The whole process is then repeated *at the same temperature* with the fats, or fatty acid, and the weights are compared. Butter and solid fats are usually weighed at 100° F. = 37·8° C. The fat is deprived of solid impurities

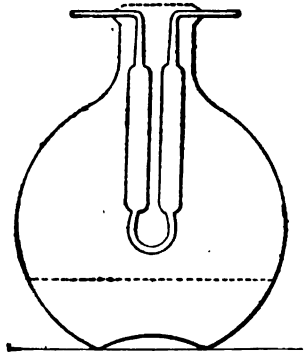


FIG. 20.—SPECIFIC GRAVITY VESSEL.

by melting in the water oven. It is allowed to cool down to 95° F. (35° C.), and transferred to the Sprengel tube in the manner described, and immediately plunged into a beaker containing water at 103° F. (39·5° C.). Thus within short limits the fat is being heated by water which is cooling, and an appreciable period of rest is obtained, and that at 100° F. (37·8° C.), during which adjustments of the quantity can be made. The position of the meniscus formed can be adjusted to the mark by applying either a small roll of blotting-paper, or the end of a glass rod which has been dipped in the oil, to the capillary end of the tube. Finally, two glass caps are put on the ends of the tube, and it is ready for weighing after drying and cooling. The net weight of the fat as determined is divided by the weight of the water in former experiment at same temperature, the quotient is the actual density of the fat at 100° F. (37·8° C.). With care, concordant results accurate to the fourth decimal place can be obtained by this method, but it is laborious, and for ordinary practice the gravity bottle will be found more convenient. It is provided with a counterpoise, and for fats can be used in substantially the same manner as the Sprengel tube.

It is also frequently very desirable to determine the "actual density" of the fatty acid, since so many fatty oils occur in commerce in a state of partial rancidity, that the density of the oil itself is not unfrequently affected thereby. If, however, the whole be converted into fatty acids, more constant and reliable results are obtained.

Further information regarding methods of determination of specific gravity, more especially as relating to oils, will be found in the chapter devoted to lubricating oils. Viscosity also, which while useful perhaps as a test of genuineness, is of more importance as indicating the value of any particular oil or mixture for reducing friction and bearing weight, will be treated under the same head.

MELTING (OR SOLIDIFYING) POINT OF THE FATTY ACIDS.

In Dalican's work (*Moniteur Scientifique*, Paris, 1868), minute instructions are given for testing the value of tallow, oils, &c., for candle-making and soap-boiling. It is stated that this method has been adopted as a standard by the tallow-melters, brokers and candle manufacturers of Paris, and it is claimed for it that it gives absolutely concordant and reliable results. The following is a summary of the process.

In an enamelled basin of at least 1 *litre* capacity, 50 *grm.* of the tallow (or oil) is heated till it begins to give off vapour [about 392° F. (200° C.)]. While this is heating, a mixture is made of 40 *c.c.* of pure caustic soda solution at 65° Tw. (35½° B.), and 30 *c.c.* of alcohol 40° Cartier or 95°–96° Gay-Lussac (sp. gr. 0·815). This mixture is added gradually to the hot tallow, the whole being well stirred, until a solid mass is formed; 1 *litre* water is added, and the whole is boiled for 45 minutes. The soap solution is then decomposed by the addition of 60 *c.c.* of sulphuric acid at 41° Tw. (24½° B.), and the whole is boiled until the fatty acids are perfectly limpid, and free from clots.

To perform the "titration," which consists in a very careful determination of the melting-point, a glass tube, 10–12 *c.* long and 1½–2 *c.* wide, is filled ⅔ full with the fatty acids, melted at as low a temperature as possible, and the tube is suspended in a flask by a perforated cork. The bulb of a delicate thermometer, whose stem is divided into fifths of a degree C., is placed in the centre of the mass of fatty acids, the thermometer being suspended for convenience of manipulation and observation. When the fatty acids begin to crystallise, a rotary movement, thrice to the right and thrice to the left, is given to the thermometer. During this operation, the thermometer falls slightly and then *rises again to a point at which it remains stationary for at least 2 minutes.* This

is the degree which is accepted as the "titre" of the tallow, and is sometimes called the melting-point, but is really the point of solidification of the fatty acids.

Degrees C.	Percentage of		Degrees C.	Percentage of	
	Stearic acid.	Oleic acid.		Stearic acid.	Oleic acid.
40·0°	35·15	59·85	45·5°	52·25	42·75
40·5°	36·10	58·90	46·0°	53·20	41·80
41·0°	38·00	57·00	46·5°	55·10	39·90
41·5°	38·95	56·05	47·0°	57·95	37·05
42·0°	39·90	54·10	47·5°	58·90	36·10
42·5°	42·75	52·25	48·0°	61·75	33·25
43·0°	43·70	51·30	48·5°	66·50	28·50
43·5°	44·65	50·35	49·0°	71·25	23·75
44·0°	47·50	47·50	49·5°	72·20	22·80
44·5°	49·40	45·60	50·0°	75·05	19·95
45·0°	51·30	48·70			

N.B.—The range of this table is between 104 and 122° F.

From the titration so obtained, the percentages of stearic and oleic acid in the original tallow may be deduced from the preceding table, constructed synthetically by Dalican, from so-called pure stearic acid, and oleic acid perfectly freed from stearic, palmitic and other hard fatty acids. In the table, 1 per cent. is allowed for loss by water and impurities, and 4 per cent. for loss by glycerin, contained in the original tallow.

It is scarcely necessary to add that, if the original fat be accurately weighed, the operations carefully conducted, and the fatty acids [freed from water by exposure to at least 248° F. (120° C.)] be then weighed, it will be seen at once whether the original tallow contained any undue impurity or fraudulent admixture. 50 *grm.* pure tallow should give 47·5 *grm.* fatty acids.

For various other commercial tests, Dalican's book may be consulted with advantage.

There are various other methods of taking "melting" or "fusing" points, but none so accurate as Dalican's. In some cases capillary tubes are used, which are filled with the fatty acid for about $\frac{1}{2}$–$\frac{3}{4}$ in. of their length, immersed in water with their ends downwards, and gradually heated in a water-bath: the point at which the semi-fluid fatty acid rises in the tube, is considered to be the melting-point. Several determinations may be made simultaneously by this method. An apparatus suitable for a third method is shown in Fig. 21. The fatty acid is in the inner tube B, surrounded by water, which is gradually heated over a lamp; temperatures are observed by thermometers C, D—C being also used as a stirring-rod. Another mode is to dip a small loop of platinum wire into the melted fatty acids, and, after withdrawing it with an adherent bead of fat, to allow the latter to cool, and then immerse it in water which is gradually heated; a sudden increase in the transparency of the bead denotes the melting-point. Determinations by this method are usually higher than by others.

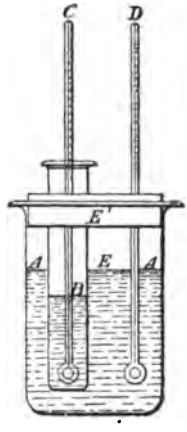


FIG. 21.—MELTING-POINT TESTER.

Whatever method be chosen, it should be adhered to, since the results by different methods cannot be compared. Moreover, long experience has convinced the writer that no observations on the so-called melting-point of neutral fats are of any practical value, since such a variety of circumstances are likely to interfere with its accurate determination.

IDENTIFICATION OF OILS IN MIXTURES.

The next subject claiming attention is the identification and testing of oils, especially when mixed, a point of the

greatest difficulty, and one which eminently requires experience. It does not, like the subject just finished, rest on a definite chemical basis; and although many processes have been from time to time advocated, none has really stood the test of repetition by other hands. The peculiarity of oils is that one analyst may have methods which may and do give fair results in his own hands, but which, repeated by others without his special experience, become not only inaccurate, but positively misleading. It cannot be too clearly impressed upon the would-be acquirer of information on this subject, that, in the present state of chemical knowledge, there are no decided and definite tests for each kind of oil, as there are for each kind of mineral substance, and that, in arriving at a conclusion as to the composition of any oil or mixture of oils, the analyst can only be guided by what may not inaptly be described as "circumstantial evidence," several tests indicating a balance of probabilities in one direction. An essential point in setting about the study of oils is the possession of a set of really genuine standard samples; this is very difficult to procure, as the oil trade is so permeated by the principle of admixture, that the refiners have too often good reason to shun any attempts to render its detection more easy.

The first step is to train the nose to distinguish between certain main groups. To do this, take some oil in a small flat porcelain basin, warm it up to about 300° F. (142° C.), and observe the smell. Then, as soon as sufficiently cool, rub some into the palm of the hand, and again smell. A little practice will thus permit the easy detection and distinction between (1) marine animal oil, (2) terrestrial animal oil, (3) vegetable oil, (4) mineral oil. The odours of these four classes are entirely *sui generis*, and it is safe to pronounce on the main question by this test. The marine oils have all the repulsive fishy odour in various degree, the sperm requiring most practice; the other animal oils have all the peculiar sourish smell of cooking animal fat, soon learned by experience; the vegetable oils, on the other hand, have a more or less sweetish odour, and practice will even enable most of

them to be named. Mineral oils of the paraffin class are easily recognisable, and so is the peculiar odour of any fatty oil which has been distilled.

A large number of tests are known as "colour tests" and depend upon the production of certain colours when different reagents act upon pure oils and mixtures thereof, and their general behaviour under these conditions. They are, however, very liable to be interfered with by the presence of free fatty acids in the oil. The usual mode as first brought out by Chateau in 1861, is to allow 5 drops of the reagent to act upon 10 drops of the oil in a white capsule, and to observe the results at the end of short intervals. The reagents usually employed are :—Sulphuric acid, phosphoric acid, nitrous or nitric acid, zinc chloride, barium polysulphide, stannic (tin) chloride, and mercuric nitrate, with or without sulphuric acid.* With the exception of the first they are of very limited utility.

A more refined colour-test consists in observing the absorption-bands of the fixed oils by means of the microspectroscope. No oils of animal origin give definite bands, while in many vegetable oils the absorptive bands, due to chlorophyll and other impurities, are well marked, especially those near the Fraunhofer line B. Hence rape, olive, or linseed oil may readily be detected in sperm, cod, or lard oil. Some vegetable oils, however, e. g. castor oil, have no definite bands of this character. To this may be added a reference to the amount of information which may be derived from examining, by polarised and by ordinary light, under a microscope, the manner of crystallisation of thin layers of fatty acid mixtures allowed to cool between two pieces of glass pressed together; some very remarkable results of this method were shown by Price's Candle Co. at the Paris Exhibition of 1878.

"Maumené's test for oils by observing the rise of tempera-

* A full description of these tests, from the pen of Dr. Muter, will be found at pp. 1467–76 of 'Spons' Encyclopædia of Manufactures'; also in vol. ii. of Allen's 'Commercial Organic Analysis.' But Allen has more recently pointed out the fallacious nature of certain of them.

ture produced on mixing 50 *grm.* of the oil with 10 *c.c.* of sulphuric acid is a useful test dependent on the chemical constitution of the oils examined, and when carefully conducted, it gives remarkably constant indications. The following are essential conditions of success:—(1) Always to use an acid of exactly the same sp. gr., and to preserve it most jealously from the air. (2) To bring the oil and acid to exactly the same temperature before commencing an experiment. (3) To mix the oil and acid thoroughly by means of a thermometer, stirring the whole time, and not reading off the temperature till the mercury begins to fall. (4) To work with the same apparatus, and to prevent loss of heat by enclosing the beaker in a non-conducting substance. In the case of the vegetable oils, the rise of temperature increases very regularly with the drying tendencies of the oil—olive oil developing about the least heat, and linseed oil the most." *

A reagent frequently employed in the examination of olive oil is a saturated solution of mercurous nitrate which, when agitated with the oil, solidifies it in more or less time, and to a greater or less extent, according to its purity.† Various modifications of this, known as the elaïdin test, have been from time to time proposed, but none of them is very satisfactory. They all depend on the property of olein and oleic acid of yielding isomeric bodies of comparatively high melting-point under the action of nitrous acid. The process has even been proposed for operation on a manufacturing scale, but the compounds thus formed are very unstable, and exhibit a decided tendency when saponified to revert to their original soft condition.

As an example of the application of the melting-point test, the familiar case of mixtures of olive oil and cotton-

* A. H. Allen, loc. cit., p. 56. According to this author, also, even with the greatest care, results are obtained by Maumené's method which defy explanation.

† The action of this salt is in all probability due to the nitrous acid which it persistently holds in sclution.

seed oil may be taken. The fatty acids of refined cotton-seed oil melt at 97° F. (36° C.), while those of pure olive oil are fusible at about 30° F. lower, varying between 60° and 70° F. (15½°–21° C.), according to whether the oil comes from the first or last portions expressed. The inference from this is obvious.

In applying this test, it is of the *utmost* importance to ensure complete saponification of the oil, and it is very desirable to precipitate the soap from its solution in water by the addition of sodium chloride, and to dissolve the soap so precipitated in a fresh portion of water before decomposing it with mineral acid. Since there are so many ways of determining melting-points, which give different results, it is undesirable to quote actual figures here, or to do more than point out the method, and the importance of each operator keeping to one mode of working, and one system of recording his observations. Dalican's method, described above, is the one usually adopted for determining melting and solidifying-points; and its chief disadvantage is that it involves the preparation of a considerable amount of the fatty acids.

A test for the presence of coconut or palm-kernel oils, in presence of any other vegetable or animal fatty oils, depends upon the quantity of salt required to separate or precipitate their soaps from solution in a given quantity of water. It may be thus applied:—To 10 *grm.* of the pure fatty acids is added aqueous solution of caustic soda equal to 1·25 *grm.* soda (100 per cent.), in a beaker of 100–150 *c.c.* capacity, previously tared. The whole is then boiled, and water is added until the contents of the beaker weigh 50 *grm.* at 212° F. (100° C.). At this point, a saturated solution of sodium chloride is run into the beaker from a burette, and the whole is stirred and boiled over a gas flame. It will be found that while only about 8–10 *c.c.* of the solution are required with ordinary oils, coconut oil will require more than 50 *c.c.*, and mixtures of the two will take proportionate amounts to separate the soap. By keeping the solution just boiling, constantly stirring, and adding the sodium chloride

gradually, the exact moment of precipitation of the soap may easily be observed.

A somewhat refined method of differentiating fatty bodies is due to Koetstorfer, and is founded upon the determination of their "saturation-equivalent," or the exact quantity of alkali necessary to saponify them. About 1·5 to 2·5 *grm.* of the fat is treated with 25 *c.c.* of the ½ normal alcoholic potash described on p. 90; when saponification has taken place, 1 *c.c.* of an alcoholic solution of phenol-phthaleïn is added, and the liquid is titrated with ½ normal hydrochloric acid. A blank experiment is then made by titrating 25 *c.c.* of the alcoholic potash alone, and the difference in the volumes of acid used gives the volume of potash solution neutralised by the fat, each 1 *c.c.* corresponding to 0·02805 *grm.* of potassium hydrate, whence the saturation-equivalent is easily calculated. The following are a few examples:—

—	*Grm.* of Potassium hydrate per 1000 of Fat.	Average Saturation-equivalent.
Tallow	196·8	285·1
Lard	195·6	286·8
Coconut and palm-nut oils	270–275	205
Butter fat	233·4–221·5	247·1
Olive oil	191·8	298·2
Rape oil	175·0	320·6

For the detection and estimation of rosin in admixture with fats and fatty oils, the determination of the specific gravity of the fatty acids will be found a very valuable guide. The greater the proportion of rosin, the higher is the sp. gr. Since, however, some of these fatty-acid mixtures are solid at 100° F. (38° C.), it is desirable to adopt a higher temperature, say 212° F. (100° C.), as a standard at which to perform the operation. As in every other case, unknown samples presented for examination must be compared with samples of known composition.

Further methods of estimating rosin in the fatty acids derived from soaps, will be found in Chapter XI., which is devoted to the valuation and analysis of soap.

Another process, applicable to the same problem, as well as to similar ones, consists in making an "ultimate organic analysis" of the mixture, and determining by direct combustion the proportions of carbon and hydrogen in the sample, and of oxygen by "difference." Such an operation, however, can only be conducted by a skilled chemist, and cannot be described here.

A comparatively new but apparently very accurate method of examining oils and fats is founded on the differences in their power of absorbing bromine. Details will be found in the 'Journal of the Society of Chemical Industry,' ii. 435–438. The test may be applied in one of two ways, according to whether it is desirable that water should be present or not. In the dry method, as worked out by Mills and Snodgrass, carbon bisulphide is the common solvent of the bromine and the substance to be treated, and the process is carried out as follows:—

"The substance having been first dried by heat, or by filtration through a quantity of dry paper scrap (which is usually quite sufficient), is dissolved in the bisulphide so as to make a solution of 10 per cent. or less strength. A definite volume of this solution is then placed in a narrow-mouthed stoppered bottle of 100 *c.c.* capacity, and diluted with more bisulphide to about 50 *c.c.* A deci-normal solution of bromine in the bisulphide is then run in gradually and in successive portions, with agitation, until the colour of the free bromine remains permanent for ¼ hour. During this operation direct sunlight must be carefully avoided. At the end of that time, to 50 *c.c.* of bisulphide, in an exactly similar bottle, standard bromine is added until the tint in both bottles is the same. The number of *c.c.* used in the blank experiment is then deducted from the number in the absorption experiment, and the remainder gives the requisite datum for calculating the percentage of bromine added.

The probable error of a single determination of a percentage absorption by this process is, on the average, about 1·36; that of the mean of 3 experiments, 0·79."

Modifications of, and additions to, this method are frequently being proposed by the original authors of the process, details of which usually appear in the 'Journal of the Society of Chemical Industry.' As an example, reference may be made to the number for February 1885.

Another generally applicable method for the examination of fatty substances has been proposed by Hübl,* who employs an alcoholic solution of mixed iodine and mercuric chloride: 25 *grm.* of iodine are dissolved in ½ *litre* of alcohol 95 per cent., free from fusel oil, and 30 *grm.* of mercuric chloride in another ½ *litre* of the same. The two solutions are then mixed, after filtration if necessary, and used after 12 hours' standing; it must also be standardised immediately before or after use. About 0·2–0·4 *grm.* of oils, and 0·8–1·0 *grm.* of solid fats, is weighed off, and dissolved in 10 *c.c.* of chloroform; 20 *c.c.* iodine solution is added, and successive additions of 5 or 10 *c.c.* are made until, after 2 hours, the solution has a dark brown tint. The next 10–15 *c.c.* of a 10 per cent aqueous solution of potassium iodide are added, and 150 *c.c.* of water. The free iodine is then titrated with solution of sodium thiosulphate containing 24 *grm.* per *litre.* The amount of iodine absorbed is calculated into units per cent. of the fat, and may be conveniently termed the Iodine degree. This number appears to be tolerably constant for each oil, or class of oils, and is highest with the vegetable drying oils, as will be seen by this short list taken from Hübl's table:—Linseed oil, 158; hempseed oil, 143; cottonseed oil, 106; olive oil, 82·3; lard, 59·0; palm oil, 51·5; tallow, 40·0; coconut, 8·9.

* Dingl. Polyt. Journal, 253, 281, 1884; Jour. Soc. Chem. Ind. iii.

CHAPTER VI.

CAUSTIC ALKALI—AND OTHER MINERAL SALTS.

THE main subject of this chapter is the preparation of the caustic lye (whether of soda or potash) used in soapmaking, starting in the case of soda from the commercial product known as "saltcake," i.e. sodium sulphate. The various sources of potash, and their conversion into caustic potash, will be described. The manufacture of those curious salts which may almost be termed mineral soaps, sodium and potassium silicates, will also be briefly dealt with, and a very short notice will be given of some of the alkaline salts which, for various purposes, are incorporated with soap.

It was stated in Chap. II. that "hard" soaps contained soda as a base, and "soft" soaps contained potash. As the production of hard soaps far exceeds that of soft, it will be convenient to describe first the processes necessary to produce caustic soda lye. These may be classed under three heads, according to the starting point of the process, and they may be thus arranged in order of simplicity:—

A. By the simple solution of solid caustic soda in water.
B. By causticising a solution of soda ash with quicklime.
C. By causticising the crude soda liquor obtained from the lixiviation of "black-ash," which is produced during the conversion of saltcake into soda-ash.

From the manufacturer's point of view, the cost of these processes is in the inverse order of their simplicity, owing to the relative market value of the caustic soda, soda-ash, and saltcake respectively. Hence, almost all large soapmakers in the United Kingdom adopt the last process, which is

very slowly finding its way into America, where the second process has been in vogue for many years, even in the very largest factories. The first method is chiefly confined to small soaphouses, to makers of high-priced toilet soaps (where expense is a matter of less moment), and to those consumers, whether manufacturers or private individuals, who prefer to make their own soap.

Nearly the whole of our soda is now obtained from common salt (sodium chloride), and until a very few years ago it was practically all obtained by the Leblanc process. This consists of three stages:—(1) The conversion of salt into saltcake (i. e. of sodium chloride into sodium sulphate), by heating the former with sulphuric acid; (2) the manufacture of "black-ash," by which sodium sulphate is transforme into a crude sodium carbonate by fusion with coal and limestone; (3) the preparation of soda-ash and caustic soda in their numerous forms, from black-ash. Within the last ~~three~~ 13 or (14) four teen years, however, the efforts of Solvay in Belgium, and of Mond and others in England, have resulted in carrying out to an industrial success the laboratory reaction long known, that when carbondioxide is passed into a solution of common salt and ammonia, sodium bi-carbonate and ammonium chloride are formed:—

$$NH_3 + CO_2 + NaCl + H_2O = NH_4Cl + HNaCO_3.$$

Or, in shorter form, ammonium bicarbonate and sodium chloride in solution produce sodium bicarbonate and ammonium chloride. The former is decomposed by heat to yield neutral carbonate, the latter is, or may be, distilled with lime or magnesia, by which the ammonia is recovered, and used over again. Soda made in this way is known as "ammonia soda," and the process has been developed to such an extent as seriously to threaten the continued existence of the Leblanc method. This is not the place, however, to discuss the relative positions of the two industries; those interested in the matter will find an excellent summary of it in the Presidential Address of Walter Weldon, F.R.S.,

to the London Section of the Society of Chemical Industry (and published in its journal) in 1883. The local conditions required for the ammonia-soda process are so peculiar, and the practical difficulties of the process itself are so great, that it is not at all likely to be adopted by soap-boilers who make their own soda. The "ammonia soda," as it is called in commerce, is now being extensively and increasingly used by soapmakers, and especially by those who, from local difficulties in disposing of their tank waste (p. 137), cannot make their own soda.

It is quite otherwise, however, with the two last stages of the Leblanc process, which are well within the reach of all but very small soap-boilers, and hence a careful description will now be given of these. The first stage, viz. the production of sodium sulphate, is one which cannot be conducted well with a small plant, and it also involves, almost necessarily, the simultaneous carrying on of several other processes, such as the manufacture of bleaching powder; hence no account will be given of this, and those seeking information upon it are referred to treatises on alkali manufacture in general.

MANUFACTURE OF BLACK-ASH.

The second operation of the Leblanc process consists in calcining saltcake, i. e. sodium sulphate, with chalk or limestone and small coal, producing the impure sodium carbonate known as "black-ash," or "ball soda."

The raw materials necessary are:—Saltcake, limestone or chalk, and mixing coal, and the plant required is a reverberatory furnace. Good sodium sulphate should come out of the furnace red-hot, and present while cooling a pale canary colour (with no shade of green upon it), which disappears when the saltcake is quite cold. The lumps when broken should show no centres of undecomposed salt. The presence of reddish lumps, while proving, indeed, that the sulphate has been well fired, indicates also the presence

of a considerable amount of free sodium chloride. The salt-cake is then, technically, "weak." The following is about the composition of an average English saltcake:—

Sodium sulphate ..	96·00	Free sulphuric acid ..	0·80
Calcium „	0·90	Insoluble matter ..	0·25
Magnesium „	0·25		
Iron and alumina ..	0·25		99·65
Sodium chloride ..	1·20		

A careful manufacturer, however, will keep the amounts of both sodium chloride and free acid below those set down. The best hand-made saltcake should test 97 per cent. of sodium sulphate, and not more than 0·5 per cent. of sodium chloride and the same amount of free sulphuric acid.

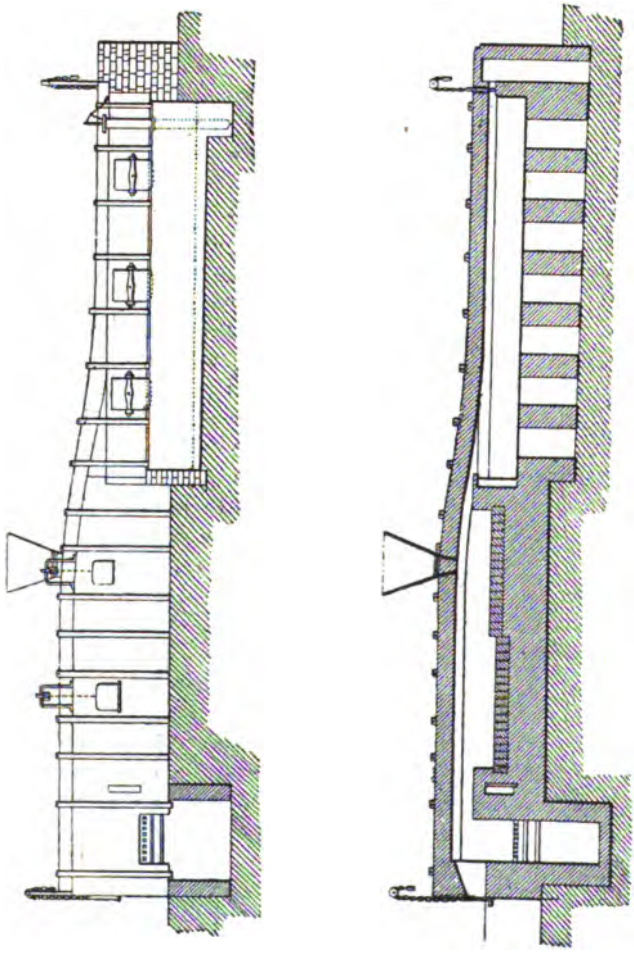
Two varieties of calcium carbonate are employed, chalk and limestone. The former is the material chiefly used in the Tyne district, for the hand-ball furnaces; the latter in Lancashire. The best chalk comes from the neighbourhood of London, Northfleet, Greenhithe, &c., and costs about 2*s.* 6*d.* per ton delivered to the works as block-chalk. "Smalls" cost about 1*s.* 6*d.* per ton. The low cost results from the custom for small coasting vessels to take it in as ballast upon their return journey. Usually containing some 12 to 15 per cent. of moisture, a portion of the chalk is dried in kilns and mixed with the fresh damp material, in quantity just enough to make the whole run well in the mill. It is then crushed between fluted rollers, or edge stones, and is ready for the furnace. If used in lumps, or wet, the sulphate in the furnace is fluxed and burned before the chalk is acted upon, and the "ball" is spoiled. Moreover, it is necessary when lumpy chalk has to be used, to put in a considerable excess, which in the lixiviating tanks gives rise to caustic soda. Good limestone, Buxton, Irish, or Welsh, as used in Lancashire and other districts, has about the following percentage composition:—

Organic matter ..	trace	Calcium carbonate ..	98·370
Silica	0·398	Magnesium „ ..	0·756
Alumina	0·135	Manganese „ ..	0·026
Ferrous carbonate ..	0·252	Calcium phosphate ..	trace

The selection of a good "mixing" coal, as it is called, is an important matter, and the quality must be kept uniform. When ignited, it should leave as small a quantity of ash as possible, and must be free from slaty and silicious matter. A bituminous gas coal mixes well. The ash should not exceed 5 per cent., nor the sulphur 0·75 per cent.

These 3 materials, in the proportion of 3 cwt. of sulphate to 3½–3¾ cwt. of chalk and about 1½ cwt. of small coal, are introduced into the ball furnace by means of a hopper, or thrown by hand upon the charging bed. The mixture varies with the judgment of the individual manufacturer, or with the state and quality of the materials; but the above proportions represent the usual charge. The furnace is reverberatory; elevation, section, and plan are given in Figs. 22, 23, and 24. The fireplace is at the end, and is about 4 ft. by 6 ft. The 2 bars that will be noticed below the grates afford leverage to the poker which is used to break up the scars or "clinkers." When these scars are removed and fall into the "cave" or fire-hole, they must be cooled with water to prevent damage to the iron. It is usual to place a water-tap in the fire-hole for the purpose. Coal is introduced through the fire-hole at the end of the furnace, which is covered with a hanging door of cast iron lined with "half-thicks." Between the fire and the first bed of the furnace a long narrow slit will be observed. This allows a current of cool air to pass continually under the bridge. One side of it is formed by what is called a "bridge-plate"—a long cast-iron slab of peculiar construction, flanged at each end and bolted into the side plates of the furnace. This keeps both bridge and furnace-bottom in their places, and prevents any fluxed material from escaping from the bed of the furnace. It is usual to give the bridge-plate sides about 2 in. high, and a lining of thin firebricks to strengthen it. The bed of the furnace is divided into 2 parts: the "working bed," that nearest to the fire, is 6 in. or so lower than the "shelf" or charging bed; the hopper in which the charge of saltcake, coal, and chalk is contained, is built into

the arch over the centre of the "shelf." Each bed is provided with a working door, closed by cast-iron covers lined



FIGS. 22, 23.—BALL FURNACES.

with half-thicks. Concerning the pan placed at the end of the furnace more will be said presently. A ball furnace

requires very careful and substantial building, to stand the heat (about 1200° F.), the wear and tear, and the contraction and expansion. The walls are about 14 in. thick, the arch 9 in., the sole 9 in., formed of 9-in. firebricks, of the best possible quality, set on end and "grouted in" with a thin mixture of finely ground fireclay and water. Below this bed a foundation of, first, concrete, and then brickwork is laid, with as thin jointings as possible. Bevelled portions of brickwork, as shown in Fig. 24, allow the workman to reach every corner of the beds with his paddle and rake. The arch goes in a nearly horizontal line over the first bed and then dips down towards the pan, so as to carry the heat well into the material. The connection between furnace and pan is formed of a bridge and air-course somewhat similar to the fire-bridge and its air-course, a large flat quarl, which projects some 4 in. over the edge of the pan, preventing the flames from coming into contact with the iron. The whole erection of furnace and pan is firmly bound up by strong iron binders running over upright bars set into the ground or foundation. The outside of the furnace is usually cased with plates of cast iron, as shown in Fig. 22, but in Lancashire it is customary simply to pass strips of metal

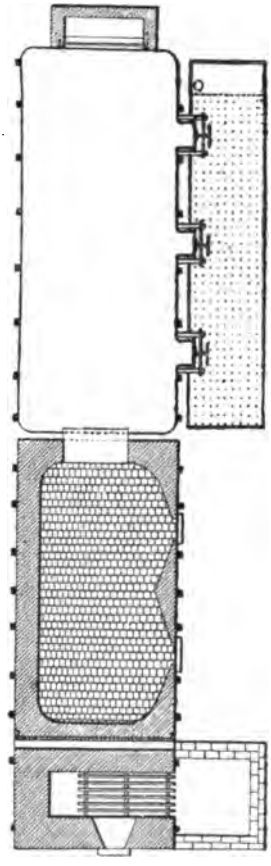


FIG. 21.—BALL FURNACE.

behind the binders. The bricks used in building a ball furnace, and especially in the beds, must be "hard burnt," and as free as possible from silica—to prevent the formation of sodium silicate. The fireclay must be as well ground as possible so that all joints may be kept fine. The charging by hopper is a great improvement over the old custom of throwing the materials down in front of the charging door and shovelling them in by hand. Not only is a considerable saving of labour effected, but less cold air is admitted into the furnace. A fresh charge is always kept in the hopper to lute it, the simple withdrawing of a slide causing the materials to fall down upon the shelf of the furnace.

It has been said that the wear and tear of a ball furnace is very great. The working bed requires renewal about every 3 months, the arch immediately over the fire lasts about the same time, while the whole furnace, except plates and foundation, requires reconstruction about once in every 3 years. With inferior workmanship in the construction, or inferior materials, the life of a furnace is much shorter than 3 years, and the renewals from time to time of the several portions mentioned are very frequent indeed.

The placing of a pan at the end of the ball furnace, as set forth in the drawings, is simply a matter of convenience and economy, to utilise the waste heat of the furnace in concentrating the black-ash liquors, and it is of more use to the ordinary alkali manufacturer who is going to make soda-ash, &c., from his black-ash liquors, than to the soapmaker who is going to causticise them at once. The latter may with advantage use the space and waste heat thus available for drying the calcium carbonate mud, which is the result of the operation of causticising, in order that it may be used in the furnace again instead of a portion of the limestone.

The balling operations are as follows:—The required quantities of chalk, saltcake and small coal are weighed off and introduced into the furnace—upon the *back* bed—by some such means as has been described. The workman with his "slice" then spreads the charge over the bed so

as to thoroughly expose every portion to the action of the flames, and shuts down the door. After a short time the charge—already called a "ball"—is raked up, half of it transferred to the bed nearest the fire, and the other half again "spread." This splitting of the ball is not a universal method of working, but is upon the whole preferable. Again the doors are closed and the split ball is exposed to the fluxing heat for about 10 minutes. The second half is now transferred to the working bed, and the really hard labour of the ball furnaceman begins, hardly ceasing until his ball is drawn. As the materials begin to soften and flux —the saltcake first—every portion must be continually turned over so as to get an even fusion, and prevent any portion being burned. This is done with the paddle, and requires great experience, strength, and judgment on the part of the workman, as his materials are constantly varying, and, technically speaking, will "stand more fire" and need more fining-down at one time than another. As soon as the fused mass begins to get stiffer, and the jets of flame, or "candles," begin to die down, the ball furnaceman takes his rake—the heavy cast-iron head about 10 in. by 7 in.—and after thoroughly mixing up every portion of the ball, draws it out as rapidly as possible into a wrought-iron barrow, or "bogie," placed under the furnace door, and just overlapped by the doorplate. All this finishing and drawing must be timed to a nicety, and calls into practice the full skill of the workman. If the ball be drawn a shade too soon, it is "green," and certain to contain undecomposed sodium sulphate; if left for a moment too long exposed to the heat, it is burned, and solidifies into a close hard mass, difficult to break up and lixiviate. A badly judged mixture is at once apparent at the finishing of the ball. If too little coal has been used, the whole mass remains soft; if too little chalk, it becomes thoroughly stiff and is difficult to draw. It takes about 40 minutes to dry, work and draw a ball. A fresh charge is introduced upon the shelf a few minutes after transferring the second half of the previous ball to the

working bed, and, after drawing, this part of the furnace is left empty for 10 minutes or so, to get up a thorough heat again, almost a white heat being required to flux rapidly. After the ball has been raked out into the bogie, it is left for a short time to cool and solidify, the "candles" or "pipes" (jets of burning carbonic oxide) rapidly dying out, and the mass assuming a creamy brown appearance; it is then wheeled away and tipped up in convenient contiguity to the lixiviating tanks.

The amount of work got out of a ball furnace varies with all different circumstances and mixtures, but, as a rule, a workman can draw 9 balls in an 8 hours' shift, well worked and fired, and weighing about 4¾ cwt. each.

The exact nature of the changes wrought in the ball furnace is still but imperfectly understood. For a full description of all the chemical theories which have been from time to time advanced, the reader is referred to the many papers that have been published upon the subject. The simplest view is, that first the sodium sulphate is reduced to sulphide by the action of the coal, and that then a mutual decomposition takes place between this substance and the calcium carbonate—sodium carbonate, carbon dioxide, and a mixture of calcium sulphide and oxide being produced. The analysis of black-ash is not only very intricate on account of the number of constituents, but is also exceedingly uncertain, from the variety of the materials used and the circumstances attending sampling and testing operations. The following, however, is given in the article Alkali in 'Spons' Encyclopædia' as representing the composition of a good and well-worked ball:—

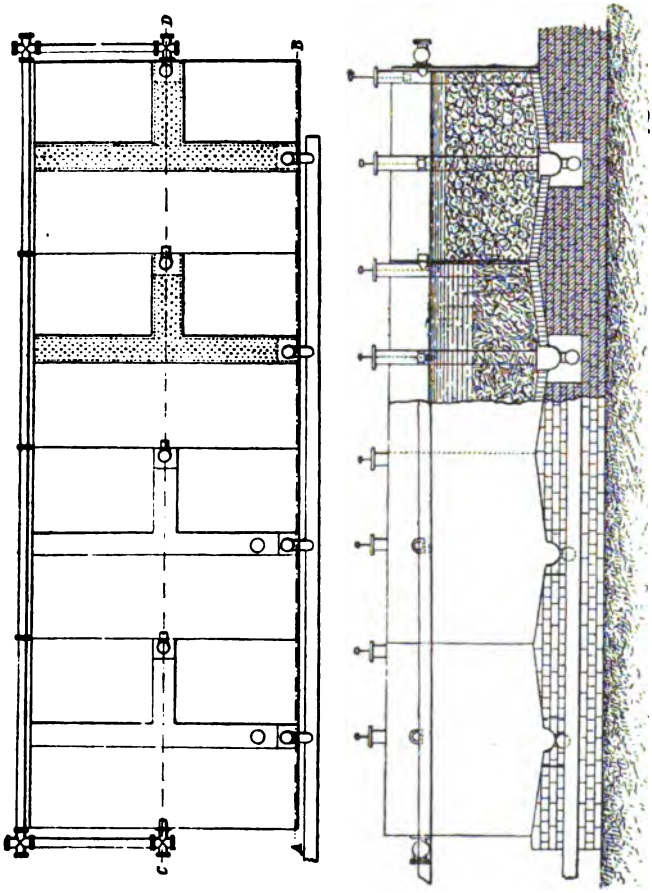
Sodium sulphate	1·00	Magnesia	0·50
„ chloride	1·50	Iron, water, and alumina	6·00
„ carbonate ..	39·00	Silica	1·70
„ silicate	trace	Sand	2·00
„ aluminate ..	trace	Carbon	4·00
„ cyanides ..	0·50	Other lime compounds ..	3·00
Calcium carbonate ..	4·00		
„ oxysulphide ..	35·00		98·70
Lime	0·35		

LIXIVIATION OF THE BLACK-ASH.

The next process is to extract the sodium compounds from the black-ash by treating the balls with warm water. It will be noticed by referring to the analysis given that about one-half of a ball is soluble and the remainder insoluble—the latter consisting of various impurities, but chiefly a mixture of different compounds of sulphur, calcium and oxygen. For the purpose of lixiviation, the balls are broken into pieces and thrown into the series of tanks, shown in Figs. 25, 26 and 27. Water at about 100°–110° F. (38°–43° C.), and the second liquors, are then run upon them, the soluble compounds are drawn off to the settlers, and the insoluble residue is thrown out. During the process of breaking up, the quality of the balls may be judged by an experienced eye almost as correctly as by complete analysis, and the careful attention of the manufacturer should be specially and unremittingly devoted to this point of review. The interior of a ball should present a clear, steel grey appearance, well honeycombed. It should break readily with a sharp ring, preferably splitting right down the centre. The outer crust should not be loose, nor too well defined, and lumps of undecomposed saltcake should be conspicuous by their absence. A pinkish shade shows a "green" ball, a dull red a burned one. Irregularity of appearance, with white lumps and dark patches, shows want of work; a general soft "mushy" character, an ill-judged mixture, or too long exposure to the air. The exact amount of harm which a ball receives by lying too long before lixiviation is a matter of doubt. If put into the tanks too hot, the temperature of the water is raised too high; if left upon the ground more than 48 hours or so, a certain amount of decomposition, with oxidation of the lime compounds, takes place. As a general rule, 12 hours may be taken as the best time for a ball to lie before being tanked.

A description of the older apparatus for lixiviating

black-ash is only interesting to the alkali antiquarian. The ingenious method at present adopted was originally the invention of Shanks of St. Helens, and leaves little to be



FIGS. 25, 26.—LIXIVIATING TANKS.

desired. It depends upon the different specific gravities of the water and liquor. The tanks vary in size with the experience and judgment of different manufacturers. Good

dimensions may be taken to be 10 ft. long by 8 ft. wide and 7 ft. deep. They are usually arranged in sets of 4—4 tanks of the size named being sufficient for 3 hand-ball furnaces, or a decomposition of 60 tons of saltcake per week—and are formed by placing partitions in one long tank. The sides, ends and bottom are formed of ⅜-in. iron plates, well riveted together, with angle-irons at all the corners. The bottom is sometimes flat, and sometimes assumes for each tank the shape shown in the drawings, sloping down to a drainer or "well," which runs along the centre of the tank. In either case a 4½-in. lining of bricks on edge is given to the bottom, leaving a cross drainer, as shown in Fig. 25. Over both longitudinal and cross drains are laid loose sheets of iron, well perforated. In each drainer, reaching just below the false bottoms, are fixed 2 "jugs," one of which communicates with the next tank and the other with a spout running along the whole range of pipes, which conveys the strong liquor to the settlers.

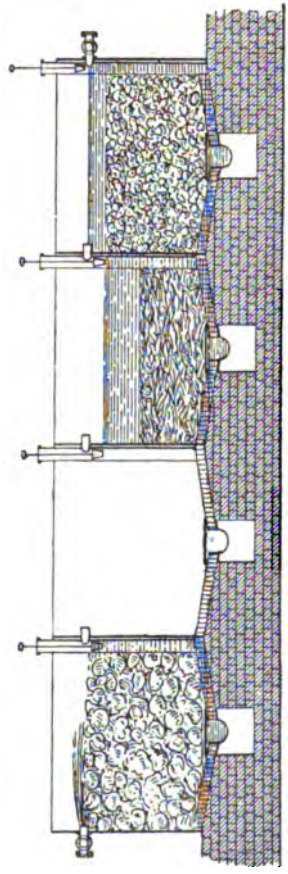


FIG. 27.—LIXIVIATING TANKS.

These jugs consist of metal pipes 3-in. bore in the lower part, widening to 4 in. in the upper part—shaped in fact like a pump. By means of a plug and seat arranged just below the

outlet pipe, or "nose," communication with the neighbouring tank or settlers can be made or cut off at will. The outlet pipes of the jugs along the front of the tanks—those by which the strong liquor runs to the settlers—are placed slightly below the level of the communications between the tanks. By a pipe running back from the fourth to the first tank the whole operation is made continuous, each one becoming in turn the "strong" tank, the intermediary and the "weak," or exhausted tank. Water is supplied to the surface of the tanks by any convenient apparatus, and is heated, *before it touches the liquor or balls*, by waste or other steam. Some manufacturers turn the steam direct into the tank, a method which causes loss through the temperature of the tank at that particular spot being raised too high, so that the sulphides are dissolved. Finally, in the drainer of each tank are fixed a pipe and cock to carry off the waste liquors.

The plan of working is as follows:—The tanks are filled with lumps of black-ash—not too large—to within about 1 ft. of the top, a layer of dry ashes being placed upon the bottoms. Water heated to about 100° F. (38° C.) is then run on, which, percolating through the mass of black-ash, rises up the jugs, and that one which communicates with the settlers being open, finds its way out in the shape of strong soda liquor. At first this liquor will test about 50° Tw. (29° B.), but the strength speedily advances to 55° Tw. (31° B.) or even 60° Tw. (33° B.), and then rapidly falls down to 40° Tw. (24° B.). The plug is then placed in its seat, and the tank is left to itself for a while. After ¼ hour or so the plug is withdrawn, and a second "running" of liquor is taken off, now testing up to 48° Tw. (28° B.) or so. Each tank should bear a third tapping, the liquor never being allowed to go to the settlers below 38° Tw. (23° B.). This outlet pipe is then closed, and the communication between the first and second tanks is opened. The liquor from the first tank flows over, percolates through the balls with which the tank is filled, and is drawn off to the settlers in the manner described. In

the meantime a steady flow of water upon the balls in the first tank is kept up. This operation is repeated with all 4 tanks. By the time the last is reached, a sample of the liquor *drawn from the jug* of the first tank will be found to test not more than 1°–2° Tw. (0·7°–1·4° B.) showing that all the soda is, practically, dissolved out. The water is then turned upon the second tank, the first being shut off. The spent liquors are drawn off through the pipe at the bottom and run away, leaving a mass of insoluble residue—tank waste—about half filling the tank. This is shovelled out, the drainer is cleaned, a fresh layer of ashes is sprinkled over the bottom, and the tank is ready to receive a supply of broken ball and the liquor to be dissolved by the returned liquors from the fourth tank. Sometimes the weak tank is "run down," as it is called, to 0° Tw.; but between 2° and 0° the sulphides dissolve more rapidly than the sodium carbonate, and spoil the liquor. Fig. 25 gives a plan of the tanks, showing the bottom drains and false bottoms; Fig. 26, elevation and sectional elevation through the line A B; Fig. 27, a section through the line C D. The last drawing shows a set of tanks as at work. No. 1 tank is just filled with black-ash, and is receiving the liquor running round from No. 4. No. 2 is empty, No. 3 is spent, No. 4 is about half through its work.

The working of the tanks is an operation requiring considerable care and judgment, much of the success of the after processes depending upon the securing of good liquor. The most important points are, to keep the temperature as low as possible; to take off the strong liquor speedily, that it may be kept from contamination; and not to allow the strength of the liquor running to the settlers to fall below 38° Tw. (23° B.). With regard to the first point, the water should not be run upon the tanks hotter than 90° F. (32° C.) in summer, and 110° F. (43° C.) in winter. The temperature of the mass in the tanks has always a tendency to rise, owing to the hydration of the lime and other chemical reactions going on. If the liquors show any greater heat than 150° F.

(65° C.), it is safe to conclude that the water has been run on too hot. Both strong and weak liquors, and tank waste, should be tested daily—at least once on each shift. The waste should present no lumps of undisintegrated ball, and should be of a dirty green colour. It should be tested at any rate for soda, and from time to time should be subjected to complete analysis. The amount of soluble soda should not exceed 0·15 per cent. A fresh sample will give about the following composition :—

	Per cent.		Per cent.
Calcium sulphide ..	37·0	Sodium carbonate ..	0·25
„ hydrate ..	9·0	Iron, alumina, and magnesia	7·0
„ carbonate ..	16·0	Carbon	6·0
„ sulphate ..	6·0	Silica, &c.	5·0
Sodium sulphide ..	0·5		

It is of the greatest importance to keep both the sulphide and the carbonate of sodium as low as possible. A good manufacturer will not allow even as much of these salts as set down in the above analysis, 0·25 total soda being the point to be aimed at.

The liquor that is drawn off to the settlers should be of a yellowish-brown colour and perfectly clear. It should be tested 2 or 3 times daily for sodium sulphide, to make sure that the tanks are not being overheated or the liquor allowed to stand too long before being drawn off. The amount shown should never exceed 0·75 per cent., though where the weak liquors are pumped back upon the tanks, and used over and over again in place of water—a piece of poor economy—as much sulphide as 2 per cent will be often registered. As this is simply converted into sulphate in the after processes, it is sheer loss of soda. An average tank liquor, not the best, will show about the following percentage composition :—

Sodium carbonate ..	69·0	Sodium cyanide	trace
„ hydrate	15·0	„ ferrocyanide ..	trace
„ sulphide	1·0	„ silicate	0·5
„ sulphite	2·0	„ aluminate ..	0·5
„ sulphate	7·0	Iron and alumina ..	0·5
„ chloride	3·0	Insoluble	0·5

The weak liquor, standing about 1° Tw. (0·7° B.) is, as has been stated, sometimes used over again in the place of water, but is usually run to waste. It contains very small proportions of sodium carbonate, hydrate, sulphide, hyposulphite, sulphate, chloride, silicate, and aluminate.

Various methods for utilising the tank-waste will be mentioned hereafter. Usually it is removed as soon as thrown out of the tanks, and either carried out to sea or deposited upon waste land. Some use is made of it in building walls and laying foundations, since the calcium sulphate or gypsum, which is formed from it by the action of the air, causes it to set very hard. If allowed to remain in heaps, as loosely thrown out of the tanks, the mass speedily becomes hot, even red hot. The oxygen of the air, and the moisture present, cause the formation of soluble calcium hydrosulphide, bisulphide, and hyposulphide, &c., and the presence of carbon dioxide causes an evolution of sulphuretted hydrogen, which is most offensive and injurious. Much of this evil can be prevented by spreading the waste over the ground, or building it promptly into whatever shape may be required, keeping out all ashes or substances that would tend to porosity, and beating it down carefully with shovels so as to keep out the air as much as possible. The most abiding mischief is caused by the drainage from all "tank heaps." The sulphide becomes soluble, and is washed out by rain, &c., forming a yellow liquid, which gives off a well-known nauseous odour. The yellow coating that appears upon the surface of a heap of tank-waste after exposure to the air consists mostly of soluble salts which have exuded and crystallised.

CAUSTICISING THE TANK LIQUORS.

When this is done regularly, as in a soap factory, it is usual to modify somewhat the ball mixture given above for the furnace. The proportion of mixing coal is increased, a large excess of limestone or chalk is added, and the lime mud

from the causticiser is usually worked up in the ball furnace. So the mixture may assume either of the following proportions :—

	a.	*b.*
	cwt.	cwt.
Saltcake	$2\frac{1}{2}$	$2\frac{1}{2}$
Limestone	$2\frac{3}{4}$	$1\frac{1}{4}$
Lime mud	none	3
Small coal	$1\frac{1}{2}$	$1\frac{1}{2}$

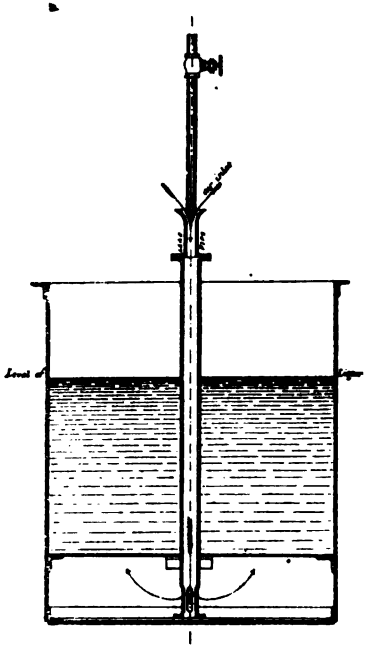


FIG. 28.—CAUSTICISER.

The admixture of lime mud varies, and with it the amount of limestone or chalk.

The tank liquors after settling are pumped into the "causticisers." These are extremely various in size and shape. Often old boilers, cut in half crosswise, are used. The best apparatus, in which the liquors are both causticised and oxidised, and at the same time thoroughly agitated, is shown in Figs. 28 and 29. But little explanation is necessary. The air oxidises the sulphides and performs the necessary agitation of the contents of the vessel, and steam helps in the agitation and heats the liquors. Steam and air are admitted below a perforated false bottom, the plan of which is given in Fig. 29. Sometimes a previous oxidation by a special blower is resorted to before the liquors are introduced into the causticiser, and mechanical agitation, by an engine fixed to the side of the vessel, is adopted, the latter addition effecting a saving of lime. A sludge valve

serves to run off the residue, or "lime mud," and the clear caustic liquors are decanted by any convenient form of syphon. Special care must be taken by the soapmaker that all the sulphides are oxidised in this operation, else his soap will be very seriously discoloured.

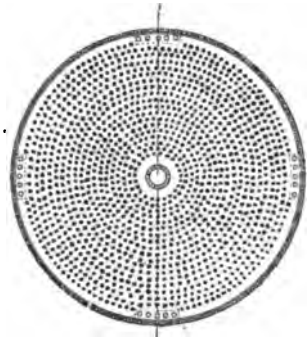


FIG. 29.—CAUSTICISER.

Before being causticised, it is usual to reduce the strength of the liquor to 20°–22° Tw. (13°–14° B.). Occasionally the reduction is carried down to 14° Tw. (9½° B.), but a liquor of 20° Tw. (13° B.) causticises as readily as one at 14° Tw. (9½° B.), and the extra amount of water simply represents an extra expenditure of fuel afterwards. Steam is blown in until a temperature of 212° F. (100° C.) is attained, and the mass of liquor begins to boil. A quantity of quicklime contained in a convenient cage, which keeps back all stones and big lumps, is then lowered into the vessel (or, perhaps more advantageously, the lime is added gradually, the lumps falling on a screen to keep back stones, &c.), and the steaming and agitation are continued until a sample of the liquor, after filtration, gives no effervescence with dilute hydrochloric or sulphuric acid. A simple view of the reaction in the causticiser is the following :—

$$Na_2CO_3 + CaO = Na_2O + CaCO_3.$$

Besides this, the sodium sulphide is converted into hyposulphite, and the alumina and silica are carried down with the calcium carbonate. About 1½ hour is required for the causticising of a batch of liquors. For the soapmaker, probably the most economical and satisfactory way is so to adjust the operation that the resulting caustic lye when cold shall be at 18°–20° Tw. (12°–13° B.). Under these conditions,

only about 3 per cent. of the total soda present is in the state of carbonate. If the lye is weaker, a fraction less soda will be lost as carbonate, but this advantage is more than counterbalanced by the need for extra salt in the soap-pan. On the other hand, the amount of soda lost as carbonate, which cannot be causticised by any excess of lime, increases rapidly above that point. In fact, under certain conditions, the reaction above quoted may actually be reversed.

The caustic lye having been thus produced, great care should be taken that it is stored for use in covered tanks, from which the air is excluded as much as possible, or at any rate where constant change in the air above the lye is prevented. If this be not done, the caustic soda will rapidly be re-converted into carbonate, by the absorption of carbonic acid from the air; normal, country air, contains about 3 parts of carbonic acid in 10,000, but the amount in town air may be more than double this. As neutral fats do not decompose sodium carbonate in the soap-copper, and rosin does so only very partially, every pound of soda thus carbonated is so much dead loss. For some kinds of soap, caustic lye at a higher density than 20° Tw. (13° B.) is useful; this may be readily made by concentrating the weaker lye in any convenient vessel. If wrought iron with riveted joints be used, the work must be exceptionally good, since the joints are very apt to leak. A ready, but expensive method of increasing the strength of weak lye, is to dissolve solid caustic soda therein.

After the completion of the operation of causticising, the contents of the vessel are allowed to settle for ½ hour or so, during which time the insoluble portions rapidly subside. The clear caustic liquor is then drawn off, a fresh lot of diluted tank liquor is run in upon the lime mud, and the causticising operation is repeated. The mud is not removed after every operation, because a certain amount of undecomposed lime is always present, and serves to causticise the next charge to some extent. After a second operation fresh water is run in upon the mud, and the whole is well

agitated. The washings are run off to dilute the tank liquors, and the mud is run out upon the "filter." This filter is usually a half boiler cut longitudinally. The bottom is paved with bricks in somewhat similar fashion to that already described when explaining the construction of the lixiviating vats, a channel being left down the centre, and the bricks only loosely put in. The actual filter is formed by layers of coke to a depth of 9 in. or so, the bottom layer composed of good-sized lumps, the top of small pieces, and a covering of coarse sand or cinders. Crushed limestone, of varying degrees of fineness, may also be used. Over the filter are laid perforated iron plates or grids, upon which the mud is run out. When a batch is spread over the grids, it is allowed some time to drain, and is then thoroughly washed with water. The drainings and washings are utilised in diluting tank liquor, and the finally hard, close mud is shovelled out of the filter and wheeled away to the ball furnaces, or mixing depôt.

An ingenious mechanical contrivance is often used to assist the draining and washing of the mud. A 2-in. iron pipe is bolted upon the bottom of the boiler, below the filter, and communicates with a small airtight tank placed upon a higher level, and connected in its turn with a vacuum pump. Upon the top of this tank is an aircock, and let into the bottom is a pipe to convey away the collected water and liquor. When a batch of mud is spread over the filter, the vacuum pump is set up, and draws away first the drainings and then the washings. These collect in the tank, and are run off to their destination. The completely washed mud should not contain above 1·5 per cent. of caustic soda. About 40 per cent. of it is calcium carbonate, 4 per cent. calcium hydrate, and 50 per cent. water.

It is highly desirable that records and tests of the whole of this process should be kept in such a way that the manufacturer can tell the result of the whole operation of soda-making, not only as regards *quality*, but *loss* during the intricacies of the process. A considerable amount of loss is

inevitable. The plant leaks in various directions, however well it may be cared for and looked after; a certain amount of soda goes away with the weak tank liquors, in the volatilisation of sodium salts, in the formation of salts—e. g. sulphide and sulphate—which do not reckon as available soda, in incomplete work in the sodium sulphate process, &c. As a rule, the average production of sodium carbonate from sulphate is not above 69 per cent.—perhaps hardly so much. In a carefully conducted alkali works, 70 parts of soda-ash of "natural strength"—i. e. 52 to 53 per cent.—should be obtained from 100 parts of sulphate. Theoretically, 75 parts should be obtained.

It is not difficult to obtain such records in the case of caustic lye, by testing average samples of each lot of liquor causticised, and keeping a register of its quantity also.

TESTING THE LYE, ETC.

It may be convenient here briefly to indicate the method of testing solutions of caustic soda, or samples of soda-ash, for the "available soda" which they contain; and for carbonic acid. Full details will be found in the various works on Alkalimetry and Analysis. The principle of the process consists in ascertaining what volume of an acid solution of known strength is required to exactly neutralise a given quantity of the lye or ash. The exact point of neutralisation is determined by the use of an "indicator," usually litmus, or, latterly, methyl orange, or sometimes phenolphthaleïn, which is colourless in acid liquids and pink in alkaline.*

The standard solution of sulphuric acid contains 49 *grs* of real sulphuric acid per *litre*, and may be made in the following way:—30 *c.c.* of the pure acid, 168° Tw. (66° Bé) is diluted with water in a beaker, and the mixture is left to stand; when perfectly cool, it is washed into a *litre* flask

* 'Journal of the Society of Chemical Industry,' i. 16, 202.

and diluted to the containing-mark. The solution is next to be tested with a standard solution of sodium carbonate, containing 53 *grm.* of the pure carbonate to the *litre*, carefully weighed and measured. 10 *c.c.* of this latter solution is placed in a beaker with a little distilled water and a few drops of the "indicator," and the acid is run in carefully and slowly until the point of saturation is reached. If more than 10 *c.c.* be required, the solution is too weak; if less, it is too strong, and it must either be strengthened or diluted, as the case may be, until 10 *c.c.* of each solution *exactly neutralise each other.* In order to ensure perfect accuracy, larger quantities of the 2 substances, say 50 or 100 *c.c.*, may be employed, when the difference, if any, will be more readily detected.

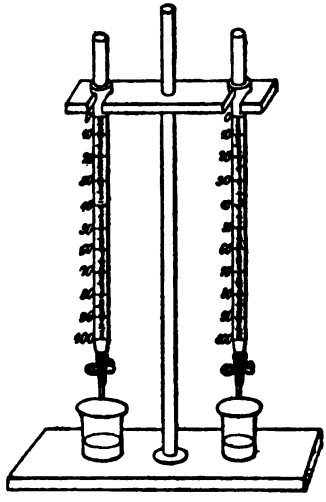


FIG. 30.—BURETTES.

If it be preferred to use caustic soda instead of carbonate, about 42 *grm.* is to be dissolved in water (about 800 *c.c.*); the above test is applied, and small quantities of water are added until equal volumes exactly correspond. All these solutions are kept in tightly-stoppered bottles.

The method of procedure is as follows:—The necessary quantity of alkali being weighed or measured, as the case may be, it is diluted with distilled water in a flask, and enough litmus is added to produce a distinct, but not too deep, blue colour. The acid from the burette (Fig. 30) is then run in until the contents of the flask have been changed to a bright red colour. In order to expel the carbonic acid,

the flask is boiled until the blue colour reappears; the acid solution must now be run in, a few drops at a time, with continued boiling, until, by the addition of a single drop, a distinct pink colour is produced. In order to obtain a very accurate result, it is well to run in an excess of acid, boil the liquid well, and then add, drop by drop, the standard alkaline solution until the liquid suddenly changes from pink to violet blue. The quantity of the alkaline solution required to effect this change is subtracted from the volume of acid originally run in, and the exact volume of standard acid required to neutralise the amount of alkali previously taken for analysis is thus determined at once. From this, the amount of soda can be calculated. The result is slightly too high, as far as the soapmaker is concerned, because the soda present as carbonate, sulphite, hyposulphite, silicate, &c., is all reckoned as available alkali.

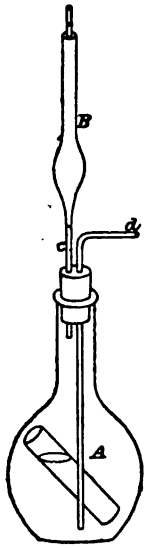


FIG. 31.
CARBONIC ACID FLASK.

Mohr recommends the use of oxalic acid instead of sulphuric or hydrochloric, because it is more readily weighed than a liquid, and because its solution may be kept for a much longer period than these without undergoing change in strength. The weight required is 63 *grm.* per *litre* of water.

The carbonic acid is usually determined directly, by the aid of the little apparatus represented in Fig. 31. The weighed carbonate is dissolved in warm water in the flask A, or the caustic lye is placed there, and a quantity of acid, more than sufficient to neutralise the alkali, is placed in the short tube in the interior. A small quantity of potassium bichromate is also added, to oxidise the liberated sulphurous acid, which would otherwise be reckoned as carbonic acid. The apparatus is

then weighed, and the tube *d* is closed by a plug of wax; the flask is tilted gently, so as to cause the acid to flow into the flask upon the carbonate. Carbonic acid is thus evolved, and the apparatus should be warmed until the evolution of gas completely ceases. When this is the case, the plug is removed, air is drawn through, and the whole is again weighed. The bulb-tube B is filled with calcium chloride to dry the evolved gas. The loss indicates the quantity of carbonic acid evolved, from which the amount of real carbonate contained in the sample may be calculated at once. The acidity of the solution, at the conclusion of the test, should be determined by adding a drop of litmus solution; if it be not acid, more acid must be added, and the operation repeated.

Another, and very usual method, of testing the causticity of lye involves 2 alkalimetrical tests, but no weighing beyond that of the samples to be tested. In one of these, the total alkali present is determined; the second is treated, while boiling, with so much barium chloride as will convert the alkaline carbonate into the insoluble barium salt; the barium carbonate is filtered off, and the alkali is determined in the filtrate; the difference between the two gives, of course, the amount of alkali present as carbonate. The results are liable to be a trifle higher than the exact truth.

Still another method is to make a direct determination by the use of phenacetolin as an indicator, which is yellow with caustic alkalies, but brick-red with alkaline carbonates and with magnesia.* Some operators, however, say that this method is difficult to work, except in the case of solutions of particular strength. In connection with this subject, it may be well to state that, commercially, soda-ash is sold by the percentage of sodium carbonate it contains, and at so much "per degree." The equivalent of sodium is usually taken as 24 instead of the correct figure 23, and of the carbonate, 108 instead of 106. Hence the correction to "English

* 'Journal of the Society of Chemical Industry,' i. 56, 57.

degrees" in the alkalimetrical table on the opposite page—the degrees upon which the carbonate is usually reckoned. The Decroizilles' degrees represent the French standard, and show the number of parts of oil of vitriol neutralised by 100 parts of the sample. This table has been drawn up by John Pattinson, of Newcastle-upon-Tyne.

In the case of nearly pure solutions of caustic soda, a very close approximation to their strength (i. e. the percentage of caustic soda) may be obtained by the observation of their density with a hydrometer, Fig. 32. These instruments are usually made very cheaply of glass, the lower end being weighted with shot or mercury. When floated,

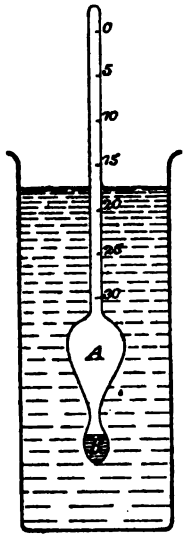


FIG. 32.
HYDROMETER.

Specific Gravity.	Per cent. of Na_2O.	Specific Gravity.	Per cent. of Na_2O.
1·4285	30·220	1·2280	14·500
1·4101	29·011	1·2058	13·297
1·3923	27·802	1·1841	12·088
1·3751	26·594	1·1630	10·879
1·3586	25·385	1·1428	9·670
1·3426	24·176	1·1233	8·462
1·3273	22·967	1·1042	7·253
1·3198	22·363	1·0855	6·044
1·3125	21·758	1·0675	4·835
1·2982	20·550	1·0500	3·626
1·2843	19·341	1·0330	2·418
1·2708	18·132	1·0163	1·209
1·2578	16·923	1·0040	0·302
1·2453	15·714		

they displace their own weight of liquid, and hence the bulk displaced varies with the sp. gr. of the liquid examined at the temperature at which the observation is made, which is indicated on the stem of the instrument. The table above (Tünnermann), gives the amount of soda (Na_2O) in solutions of varying densities.

Two hydrometer scales are in use among soapmakers,

Percentage of Soda.	Sodium Carbonate.	English Degrees.	Decroizilles' Degrees.	Percentage of Soda.	Sodium Carbonate.	English Degrees.	Decroizilles' Degrees.
30·0	51·29	30·39	47·42	54·0	92·32	54·71	85·35
30·5	52·14	30·90	48·21	54·5	93·18	55·22	86·14
31·0	53·00	31·41	49·00	55·0	94·03	55·72	86·93
31·5	53·85	31·91	49·79	55·5	94·89	56·23	87·72
32·0	54·71	32·42	50·58	56·0	95·74	56·74	88·52
32·5	55·56	32·92	51·37	56·5	96·60	57·24	89·31
33·0	56·42	33·43	52·16	57·0	97·45	57·75	90·10
33·5	57·27	33·94	52·95	57·5	98·31	58·26	90·89
34·0	58·13	34·44	53·74	58·0	99·16	58·76	91·68
34·5	58·98	34·95	54·53	58·5	100·02	59·27	92·47
35·0	59·84	35·46	55·32	59·0	100·87	59·77	93·26
35·5	60·69	35·96	56·11	59·5	101·73	60·28	94·05
36·0	61·55	36·47	56·90	60·0	102·58	60·79	94·84
36·5	62·40	36·98	57·69	60·5	103·44	61·30	95·63
37·0	63·26	37·48	58·48	61·0	104·30	61·80	96·42
37·5	64·11	37·99	59·27	61·5	105·15	62·31	97·21
38·0	64·97	38·50	60·06	62·0	106·01	62·82	98·00
38·5	65·82	39·00	60·85	62·5	106·86	63·32	98·79
39·0	66·68	39·51	61·64	63·0	107·72	63·83	99·58
39·5	67·53	40·02	62·43	63·5	108·57	64·33	100·37
40·0	68·39	40·52	63·22	64·0	109·43	64·84	101·16
40·5	69·24	41·03	64·01	64·5	110·28	65·35	101·95
41·0	70·10	41·54	64·81	65·0	111·14	65·85	102·74
41·5	70·95	42·04	65·60	65·5	111·99	66·36	103·53
42·0	71·81	42·55	66·39	66·0	112·85	66·87	104·32
42·5	72·66	43·06	67·18	66·5	113·70	67·37	105·11
43·0	73·52	43·57	67·97	67·0	114·56	67·88	105·90
43·5	74·37	44·07	68·76	67·5	115·41	68·39	106·69
44·0	75·23	44·58	69·55	68·0	116·27	68·89	107·48
44·5	76·08	45·08	70·34	68·5	117·12	69·40	108·27
45·0	76·95	45·59	71·13	69·0	117·98	69·91	109·06
45·5	77·80	46·10	71·92	69·5	118·83	70·41	109·85
46·0	78·66	46·60	72·71	70·0	119·69	70·92	110·64
46·5	79·51	47·11	73·50	70·5	120·53	71·43	111·43
47·0	80·37	47·62	74·29	71·0	121·39	71·93	112·23
47·5	81·22	48·12	75·08	71·5	122·24	72·44	113·02
48·0	82·07	48·63	75·87	72·0	123·10	72·95	113·81
48·5	82·93	49·14	76·66	72·5	123·95	73·45	114·60
49·0	83·78	49·64	77·45	73·0	124·81	73·96	115·39
49·5	84·64	50·15	78·24	73·5	125·66	74·47	116·18
50·0	85·48	50·66	79·03	74·0	126·52	74·97	116·97
50·5	86·34	51·16	79·82	74·5	127·37	75·48	117·76
51·0	87·19	51·67	80·61	75·0	128·23	75·99	118·55
51·5	88·05	52·18	81·40	75·5	129·08	76·49	119·34
52·0	88·90	52·68	82·19	76·0	129·94	77·00	120·13
52·5	89·76	53·19	82·98	76·5	130·79	77·51	120·92
53·0	90·61	53·70	83·77	77·0	131·65	78·01	121·71
53·5	91·47	54·20	84·56	77·5	132·50	78·52	122·50

those of Twaddell and Beaumé. The zero point of each is that to which the instrument sinks in distilled water at 60° F. (15½° C.). To convert degrees Twaddell into real specific gravity, multiply their number by 5, add the result to 1000, and then divide by 1000. Thus 15° Tw. = 1·075 sp. gr. The remarks in the last chapter upon the *true* sp. gr. of fats at various temperatures are applicable here also.

The following table gives the means of comparing degrees Twaddell and Beaumé* with each other, and with actual sp. gr., and also gives the approximate percentages of caustic soda and caustic potash in solutions of that sp. gr. at 60° F. (15½° C.), *provided they contain no other salts than the caustic alkalies.* It must be remembered that increase of temperature in a fluid, by increasing its bulk, diminishes its specific gravity.

Degrees Beaumé.	Degrees Twaddell.	Specific Gravity.	Per cent. of Caustic Soda.	Per cent. of Caustic Potash.
3	5·2	1·026	1·81	2·83
5	7·5	1·037	2·72	3·96
7	11·8	1·059	4·23	6·21
10	14·0	1·070	5·44	7·36
12	19·8	1·094	6·65	9·62
15	23·6	1·118	7·86	11·82
17	26·2	1·131	9·97	13·01
20	32·6	1·163	10·88	15·84
25	42·4	1·212	13·90	19·80
30	53·0	1·265	17·52	23·76
35	64·2	1·321	22·97	27·72
40	78·0	1·390	27·50	32·40

It may be noted here that for every 31 parts of pure soda required in any operation, 53 parts of pure dry sodium carbonate, or 143 parts of soda crystals, are required, and that the corresponding quantities of caustic potash and potassium carbonate necessary to do the same work are respectively 47 and 69 parts by weight. With this basis, a simple proportion sum will show in any given form the quantity of alkali required for any purpose.

* See Beaumé scales in chapter on lubricating oils.

UTILISATION OF THE TANK-WASTE.

In concluding this brief account of the preparation of caustic soda lye from saltcake, reference must be made to one objection to it—viz. that it involves the production of a very disagreeable bye-product, the waste residue from the lixiviating vats, the disposal of which is often a very difficult matter, lest it should become a source of nuisance to the neighbourhood. Hence many processes have been devised to render it innocuous, and to extract from it the large amount of sulphur which it contains. Only five of these processes have been worked industrially with any approach to success, and of these, three—viz. Hoffman's, Mactear's, and Schaffner & Helbig's (as carried out by A. M. Chance, near Birmingham)—are of no use to the mere soapmaker, since some of the materials and plant employed are only to be found in a fully equipped alkali works.

The process of Mond, patented in 1863, is the best known, and is as follows:—In addition to the tanks in ordinary use for lixiviating, 1½ times as many more are provided, or 6 extra tanks for every set of 4. In these extra tanks the waste is exposed to a strong current of air, forced through a false bottom with which the tanks are provided, for a period of 12–18 hours, depending upon the quality and texture of the material. During this oxidation process the temperature rises to about 220° F. (104° C.), and clouds of steam are given off. The surface of the waste becomes covered with bright yellow spots, their appearance being a guide to the workman as to the progress of the operation. Weak liquor from a previous lixiviation is then run upon the waste and allowed to stand for 8 hours or so, until, starting at about 18° Tw. (12° B.), the density falls to 10° Tw. (6¾° B.). Water is then run on, and the product is collected as weak liquor to be used for the next tank. This process of blowing and washing is repeated 3 times, the quantity of strong liquor obtained being less each time. The resulting liquors contain calcium sulphide, sulphite, and hyposulphite, together with small

proportions of similar sodium compounds. They are allowed to settle thoroughly in a series of tanks, and then run into a wooden vessel lined with lead, some 10 ft. in diameter, and 5 ft. deep, treated with weak hydrochloric acid from the condensers, and heated to 135° F. (57° C.) with steam. Viewed in the simplest possible form, the following reaction takes place:—

$$2CaS + CaS_2O_3 + 6HCl = 3CaCl_2 + 3H_2O + 4S.$$

The quantity of hydrochloric acid must be carefully gauged—continued till just a faint smell of sulphurous acid is apparent—and the contents of the "tub" must be well agitated continuously. The liquors are next run into tanks where the freed sulphur settles out, and is washed, dried, and melted. Ingenious though this extraction method is, and successful in so far as it deprives the tank-waste of those constituents which make it an absolute nuisance, it must be confessed that it does not repay the expenditure upon plant, and the trouble of conducting a delicate operation. It is, however, carried on to some considerable extent by manufacturers who, from the situation of their works, are specially compelled to guard against nuisance. Unless carefully watched, both in the oxidising and decomposing operations, an appreciable amount of sulphuretted hydrogen is given off. The average amount of sulphur recovered is $\frac{2}{3}$ of the total amount in the waste.

While the details of Schaffner's process differ considerably from the above, the substances employed, as well as the ultimate product, are the same; hence both are equally suitable for the soapmaker, whose choice between them must be guided by local conditions, the price of labour, &c. In Schaffner's process, the waste is piled in peculiarly-constructed heaps, and undergoes slow atmospheric oxidation, after which it is lixiviated. Hence much more land is necessary than when tanks are used.

PREPARATION OF CAUSTIC LYE FROM SODA-ASH.

This operation is very simple, all that is necessary being to dissolve the soda-ash in water, and to boil it with the requisite quantity of quicklime, conducting the operation precisely as described for causticising black-ash liquors. Every 31 parts of pure soda employed require 28 parts of pure quicklime, on the assumption that all the soda present is in the state of carbonate. In practice, of course, an allowance must be made for "grip"—i. e. unburnt limestone—and other impurities.

Solid caustic soda requires more time and heat for its solution in water than might be expected, and the solution usually needs some hours' subsidence, for the deposition of its impurities. It may be well to repeat here the caution against letting any sodium sulphide (a very common impurity) find its way into the soap-pan. The blast, or oxidising stream of air, should be continued through the lye for some time after all traces of sulphide (easily detected by a solution of any lead salt) have disappeared, since the slightly-oxidised compounds which are thus produced have a decided tendency to become reduced again to the condition of sulphide.

CAUSTIC POTASH.

Upon a manufacturing scale, the following are the principal sources of potash:—Probably about one-half of the total produce is still made from the ashes of land and marine plants; one-fourth from potassium sulphate, produced by the decomposition of the chloride by sulphuric acid, and from various potassium compounds; the remainder from beetroot molasses, "suint," or the wool of sheep impregnated with the sweat exuded from the skin, and from felspar and other silicates. Although the last-named source is as yet comparatively untried ground, it should be noted that in reality felspar and the other silicates yield, in the first instance, their potash

salts to all the other sources. The ashes obtained by the calcination of plants show great diversities in quantity and composition, due mainly to the soil in which the plants have been grown. The method of lixiviating the ash corresponds almost exactly with the tank operations of the Leblanc soda process, and the resulting liquor is simply evaporated to dryness in open iron pans, producing commercial "pot-ash." American pot-ashes are graded into 3 brands—"firsts," containing 54–58 per cent. of potash; "seconds," 48–52 per cent.; and "thirds," 35–40 per cent. If the pot-ash be thoroughly calcined, so as to burn out all the carbonaceous matters, the product is called "pearl-ash" and is nearly white. The composition of an average sample of good Canadian pearl-ash is given in the following table:—

Potassium carbonate ..	74·44	Insoluble silica, &c. ..	0·35
„ sulphate ..	13·01	Water	4·72
„ chloride ..	3·14		
Sodium carbonate ..	2·58		100·00
Soluble silica, &c. ..	1·76		

American pot-ashes contain, as a rule, more caustic potash than other qualities. This is owing to the use of lime in the lixiviating tanks; and, for many purposes, as, for example, soapmaking, where the pot-ash has ultimately to be causticised, it is a positive advantage, saving lime in the after process.

The amount of potash obtained from timber is exceedingly small, as compared with the quantity of the latter consumed. Pine-wood contains about 1·25 per cent. of ash, of which about 0·12 per cent. is potash; hence, in order to produce 1 ton of commercial pot-ashes, containing 65 per cent. of total pot-ash, about 520 tons of timber would have to be burned. The total yield from this source is 20,000 tons.

New and better methods of manufacture have to a large extent supplanted the wood process; but the industry is still a very large one. It is carried on chiefly in America, Canada, Russia, France, Italy, Poland, Belgium, and Austria; America alone supplies about one-half of the total amount

manufactured. The prices range from 20*s.* to 40*s.* per cwt. for pot-ashes, and from 28*s.* to 32*s.* for best pearl-ashes.

Of late years, however, a large proportion of the pot-ash salts which find their way into the English market are derived from the so-called "Stassfurt salts" produced from the alkaline minerals which occur in enormous quantities in the valley of the river Bode, about 25 miles south-west of Magdeburg, in Prussia.* The potash deposits, which for some time were looked upcn as an incumbrance, were first utilised in 1860; at present over a score of factories in the neighbourhood produce potassium chloride fron the raw mineral carnallite, which is a mixture of magnesium and potassium chlorides with kieserite, a magnesian mineral. The Stassfurt Mining Company's plant alone is sufficient to consume 600 tons of raw material daily.

The manufacture of potassium chloride is based on the decomposition of the carnallite contained in the raw material, in a hot saturated solution, potassium chloride crystallising out, and magnesium chloride remaining in solution. The hot solution is brought to 68° Tw. (36½° B.), diluted to 64° Tw. (35° B.), run into settling tanks, and allowed to crystallise; these crystals, once refined, are almost pure potassium chloride.

The next stages of the process are almost identical with those of the Leblanc soda process, the raw material (potassium sulphate) being obtained either from potassium chloride and sulphuric acid by the ordinary sulphate process, or by decomposing the former with magnesium sulphate or kieserite. Equal weights of potassium sulphate and finely divided limestone, or chalk, together with varying quantities of small coal, are roasted together in a reverberatory furnace, the product being an exceedingly impure potassium carbonate. When the decomposition is complete, the molten mass is raked out, broken up when cool, and lixiviated in

* A very interesting account of the whole industry, by C. Napier Hake, is to be found in the 'Journal of the Society of Chemical Industry,' li. 146–52.

tanks. The soluble potassium and sodium salts are thereby dissolved out, evaporated, and calcined in a small reverberatory furnace.

A carbonate of better quality is produced by following more closely the carbonating operation of the soda process. The liquors from the tanks are evaporated, the potassium chloride and sulphate which separate out during the concentration being skimmed off, and sawdust is thrown in. The dried salts are then removed to the carbonator and exposed to a heat at first gentle, but finally urged to dull redness. By this process the sulphur compounds are oxidised into sulphate, and the caustic potash is converted into carbonate. The chief object of the sawdust is to keep the mass of salt open. A carbonate carefully made in this manner should have about the following composition :—

Potassium carbonate	89·59	Insoluble matter	0·13
„ sulphate	3·98	Water	1·27
„ chloride	2·49		
Sodium carbonate	2·33		99·98
Soluble silica and alumina	0·19		

The manufacture of potassium carbonate from the sulphate by the methods described is a rapidly increasing industry. Something like 15,000 tons per annum are now produced, and, all things considered, it seems likely to supersede the other processes. It is much more amenable to the altered conditions of the labour market; a pure article is more readily obtained; and the soil is not impoverished by a too rapid withdrawal of the potash compounds, which must be the case where beetroot and other plants are the material operated upon.

It is of great importance to free the potassium carbonate as much as possible from soda compounds, as these considerably destroy the fine character which the potash salts give to the articles manufactured from them. In the preparation of soft soaps, for example, the potash compound should not contain above 3 per cent. of soda.

Other large sources of potash salts are beetroot molasses,

which yield about 12,000 tons a year; and "suint," or the solid residue from the sweat of sheep, which is retained in the wool,—44·5 per cent. of which residue is potassium carbonate yields 1000 tons.

The mode of preparing caustic potash lye is precisely the same as that described for caustic soda lye, *mutatis mutandis*; in causticising for example, every 47 parts of potash, or 69 parts of pure potassium carbonate require 28 parts of quicklime.

The following table by Tünnermann will be found useful:—

Specific Gravity.	KHO per cent.	K_2O per cent.	Specific Gravity.	KHO per cent.	K_2O per cent.
1·3300	33·693	28·290	1·1437	16·846	14·145
1·3131	32·345	27·158	1·1308	15·498	13·013
1·2966	30·998	26·027	1·1182	14·151	11·882
1·2805	29·650	24·895	1·1059	12·803	10·750
1·2648	28·303	23·764	1·0938	11·456	9·619
1·2493	26·954	22·632	1·0819	10·108	8·487
1·2342	25·606	21·500	1·0703	8·760	7·355
1·2268	24·933	20·935	1·0589	7·412	6·224
1·2122	23·585	19·803	1·0478	5·957	5·002
1·1979	22·237	18·671	1·0369	4·717	3·961
1·1839	20·890	17·540	1·0260	3·369	2·829
1·1702	19·542	16·408	1·0153	2·021	1·697
1·1568	18·195	15·277	1·0050	0·6738	0·5658

POTASSIUM AND SODIUM SILICATES.

Of the various salts employed for mixing with soaps, these are probably the most important, and in some aspects they may be regarded as mineral soaps.

These compounds, known also as soluble glass or water-glass, may be prepared either by the dry or wet methods. The first is usually adopted by Gossage, Crosfield, and others; it depends on the fact that, at high temperatures, silica plays the part of a very strong acid, capable of displac-

ing acids much stronger than itself at ordinary temperatures. On the clean hearth of a reverberatory furnace, 9 parts of soda-ash at 50 per cent. soda are fused with 11 parts clean white sand, or (for the potash salt) 45 parts of sand, 3 of charcoal, and 30 of potassium carbonate. The product, which is a glassy looking substance, may be sold in the dry state, or may be dissolved in boiling water; not unfrequently boiling under pressure is necessary to effect complete solution. If the solution be too alkaline, it may be boiled with rosin, or a fatty acid, or it may be treated with a mineral acid, either liquid or gaseous. Instead of sodium carbonate, a mixture of "saltcake" (sodium sulphate) and coal may be fused with sand, and the mixture be decolorised by sodium arseniate (or a mixture of white arsenic, sodium nitrate, and soda-ash); but a much higher temperature is required in this case, and the wear and tear of the furnace is very great.

For purposes where uniformity of composition is important, it is far better to employ the wet method, as is used by Ransome for artificial stone, and by Christr. Thomas & Bros., Bristol. In this case, white sand or calcined flint is put into a Papin's digester, with a solution of caustic soda at about 18° Tw. (12° B.). Steam is turned into the jacket, and maintained there at about 25–30 lb. a sq. in.; occasional samples are drawn off by a trycock, and when all trace of causticity has disappeared, steam is turned off, and the contents are "blown out" into tanks where a few hours' subsidence deprives the solution of all suspended impurity. If the flints are not thoroughly calcined, the organic matter in them reduces the sodium hyposulphite to sodium sulphide, which produces the usual discoloration in soap. It is then about 40° Tw. (24° B.) and may be concentrated, if desired, as far as 140° Tw. (59½° B.). Any mechanical arrangement that moves the flints about facilitates their solution. Made in this way, the silica and the soda bear to each other a very simple, but a very constant ratio, viz. 2 to 1, and hence great uniformity of composition is obtained, which is not always the case when soluble silicates are made in the fur-

nace. The compound is usually sold in solution at 140° Tw. (59½° B.), and should scarcely vary from this composition :—

Silica	33·00	per cent. to	32·00
Soda	16·50	„	16·00
Other soda salts	2·50	„	3·00
Water	48·00	„	49·00
	100·00		100·00

Solutions of sodium silicate, containing a larger proportion of silica than 2 to 1, cannot be concentrated so far, but are very suitable for many soaps; those containing less silica than 2 to 1 are unsuitable for all soaps, and should be carefully avoided.

SODIUM ALUMINATE.

As a detergent for mixing with soap, this substance is, perhaps, even more powerful than sodium silicate. It is chiefly obtained from cryolite, a mineral found in great abundance in Greenland, and may be readily prepared from it by boiling it with lime; cryolite, being a double aluminium and sodium fluoride, gives up the whole of its fluorine to the lime, leaving a mixture, or compound, of alumina and soda. Like sodium silicate, it is not a definite chemical compound, as will be seen by the following analyses of different samples:—

	A.	B.
Soda	43·0	44·0
Alumina	48·0	24·0
Water	9·0	32·0
	100·0	100·0

The commercial product is an amorphous white substance, readily soluble in water, in which state it may be mixed with soap, like sodium silicate.

MISCELLANEOUS SALTS.

Many other soda and potash salts are at times used to mix with soaps for various purposes. The most general are sodium chloride, sulphate and carbonate, which latter may be readily crystallised from "saltcake" and soda-ash respectively, by dissolving them in warm water, allowing the impurities to subside, and running off the clear solution into crystallising-pans. Borax is occasionally added to soap, and "salts of tartar," the commercial potassium bitartrate. In fact, almost any potassium or sodium salt may be thus employed, if its price, or the peculiar effect which it has upon the soap, render it desirable thus to add it. Only purely alkaline salts, however, can be used in this way; if a salt of any other base were mixed with soap, mutual decomposition would ensue, resulting in the formation of a soap insoluble in water. The detection of impurities in these alkaline salts comes under the usual methods of instruction in inorganic analysis, and their preparation is hardly likely to be undertaken by the soap manufacturer; hence nothing further need be said upon the subject.

CHAPTER VII.

MANUFACTURE OF HOUSEHOLD SOAPS:—THE PROCESS OF SAPONIFICATION.

BEFORE proceeding to describe the process of saponification on a large scale, where operations involving considerable mechanical, physical, and chemical knowledge are conducted with the view of producing the best possible article at the lowest cost, a short space will be devoted to instructions for making small quantities of soap of inferior appearance, which will answer well for homely purposes, for the benefit of those living far from large towns, and who may yet have on their farms or stations many of the ingredients necessary for their production.

For those who have plenty of fat or oil at command, but no alkali, the small canisters of pure powdered caustic soda and caustic potash, sold by the Greenbank Alkali Co., St. Helens, will be found very convenient. With these products, soaps can be made without any boiling. For a hard soap, dissolve 10 lb. of this soda in 4 gal. of water, and allow the lye to cool. Take 75 lb. of clean fat or oil, rendered fluid by heat if necessary, and when it feels just warm to the hand, add the lye to it in an uninterrupted stream, stirring well all the time; continue the stirring for 15 or 20 minutes, and then set aside in a warm place for a day. In this interval, the soda reacts upon the fat, and turns out its glycerin, which remains mixed with the soap. Any impurities in the fat used, such as salt or any other extraneous substance, will be apt to spoil the operation. For soft soap, use 20 lb. of this caustic potash, dissolved in 3½ gal.

of water, and mix as above with 8¾ gal. of cotton-seed, fish, or other (non-mineral) oil. For a harder soap, one or more of the gallons of oil may be replaced by 10 lb. (or a corresponding multiple thereof) of tallow.

A very firm soap may be made on a small scale from the oleic acid of candle-factories, known commercially as olein or red-oil, by heating it to about 212° F. (100° C.) and adding thereto ½ its weight of caustic soda lye at 66½° Tw. (36° B.). The combination takes place instantaneously, and it is only necessary to allow the soap to get cold, when it is fit for use. For the preparation of the lye, see Chap. VI. It need not be perfectly caustic; but if any sodium carbonate be present, the lye must be of proportionately higher sp. gr., and the vessels employed must be capacious, in consequence of the effervescence that occurs.

Hard soaps may also be made on a small scale without boiling, by adding to a mixture of 2 parts tallow and 1 part coconut oil, or of 3 parts tallow and 2 parts coconut oil, ½ its weight of caustic soda lye at 66½° Tw. (36° B.), the whole being at a temperature of 130°–140° F. (55°–60° C.); the mixture, after being well stirred, should be set aside for a day or two. Here also the presence of common salt is a serious obstacle to the combination.

Soaps that require boiling cannot be well prepared in small quantities. Those who wish to make them, however, would do well to study the description of the process on a large scale. In making small quantities of hard soap, it will be well to boil together the fat and the soda lye previously well causticised by lime, and to calculate from the table on p. 134 how much lye is necessary, taking as a basis that for every 10 lb. of fat, about 1 lb., or rather less, of pure soda (100 per cent.) is required. Combination will not take place unless the solution is quite weak, say 18° Tw. (12° B.), and when it is effected, salt may be added to separate the excess of water. The salt should be sprinkled in gradually, time being allowed for each portion to dissolve, and when a small sample, taken out on a shovel and allowed to cool,

separates into liquor and soap, enough salt has been added; the boiling should then be stopped, and the whole allowed to repose; in an hour's time, the soap may be skimmed off. Soft soap is more easily made by boiling on a small scale, since the process is the same whatever quantities of materials are employed. The reader is, therefore, referred to p. 165 for instructions under this head.

In considering the manufacture of soap in large quantities, the subject may be conveniently and naturally divided under the following heads:—

I. The apparatus and processes employed in effecting the chemical combination between the fatty matter and alkali, including in this a general description of the mode of boiling (or otherwise preparing) soaps of different types.

II. The machinery and mechanical and physical contrivances made use of to convert the chemical compound so produced into a marketable soap.

III. Ingredients and formulæ for the production of special kinds of soaps for particular purposes, including toilet soaps, manufacturers' soaps, &c.

These two last will be considered in the next chapter, but it will be convenient to treat, in this chapter, in the order of their complexity, the somewhat extensive range of subjects included under I., commencing with the simplest, and, accordingly, we find the following natural subdivisions:

I*a*. Soaps produced by the direct union of fatty acids and caustic alkali, or by the decomposition of carbonated alkali by fatty acids.

I*b*. Soaps produced by the action upon a neutral fat, of the precise quantity of alkali necessary for saponification without the separation of any waste liquor, the glycerin being retained in the soap. This class includes—*α*. Soaps made by the "cold process"; *β*. Soaps made under pressure.

I*c*. Soaps produced by the ordinary methods of boiling in open vessels, working with indefinite quantities of alkaline lye, the processes being controlled by the experience of the operator. These are again subdivided into—*α*. Soft soaps,

in which the glycerin is retained, potash being the base; β. The so-called "hydrated" soaps, with soda for a base, in which the glycerin is retained, and of which "marine" soap may be taken as the type; γ. Hard soaps, with soda for a base, in which the glycerin is eliminated, comprising 3 kinds — curd, mottled and yellow soaps.

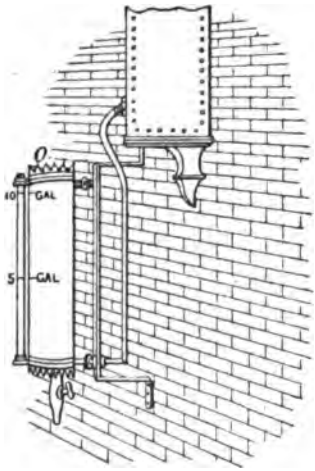


FIG. 33.—GAUGE TANKS.

It may be noted that a very large proportion of all the soaps manufactured is included in this last and most complex subdivision, since practical experience shows that, all things being considered, they are the most marketable.

Full directions for the fabrication of these several kinds will now be given, the paragraphs treating of each being numbered to correspond with the above classification.

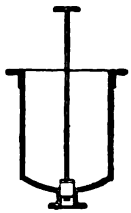
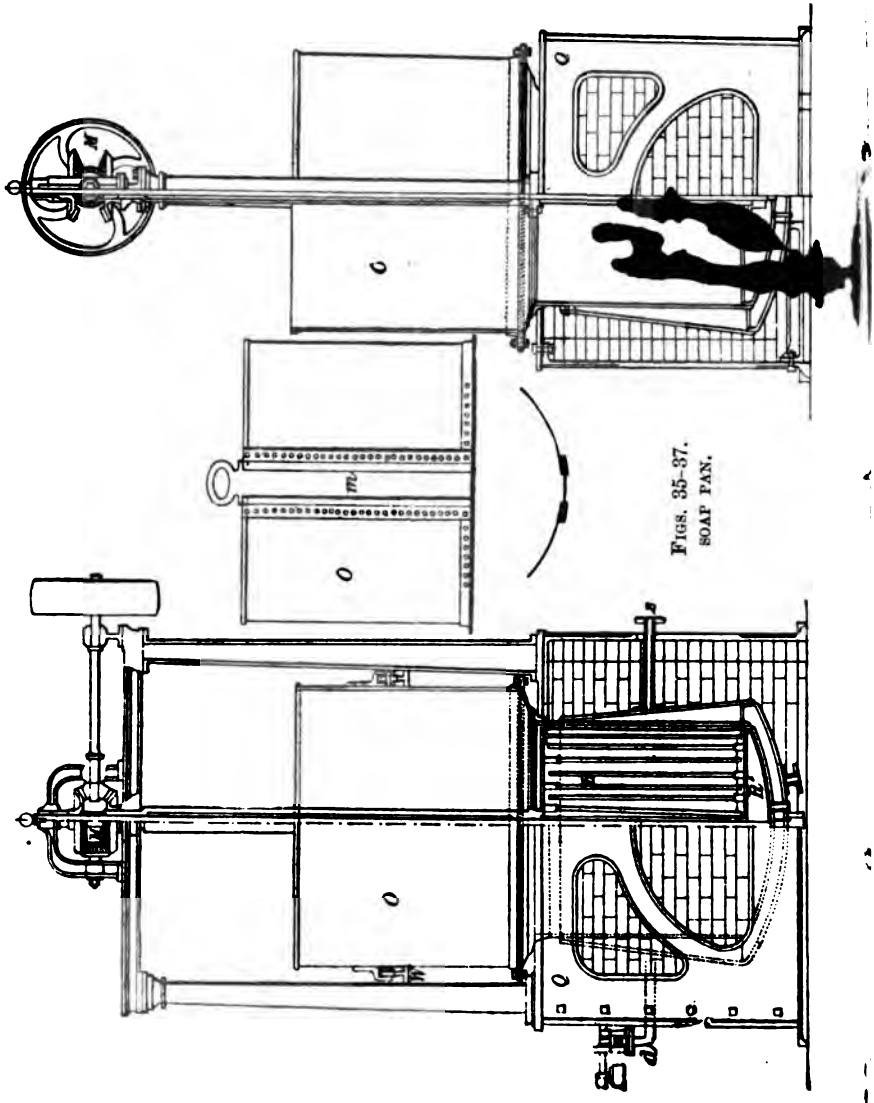


FIG. 34.—FEEDER.

I*a*. In soaps made from fatty acids, the soda is generally used in the form of a refined carbonated ash at 52° (that prepared by the Jarrow Co., Newcastle-on-Tyne, is recommended), every 100 lb. being dissolved in 160 lb. water in a lead-lined vat, and the solution allowed to settle previous to use. The store-tanks of this, and of the fatty acids employed, are connected with small gauge-tanks or measuring-tubes (Fig. 33), for the purpose of obtaining uniformity in the results by the use of exact quantities in every operation. For the delivery

of the soda solutions into the soap-pan, a special feeder (Fig. 34) is provided, closed with a movable plug, by which the flow of liquid may be regulated at discretion; a perforated rose-spout may be advantageously placed under the exit-pipe.

The soap-pan in which the operation is conducted, shown in Figs. 35, 36, 37, is jacketed, the inlet-pipe being at Z, and the steam is either superheated or used at a pressure of 75–80 lb. Above the pan is a movable curb O, with slide at *m*, necessary to give room for the intumescence caused by the liberated carbonic acid; a wheel arrangement W enables it to be readily drawn aside on a railway behind the columns N, which support the gearing M. This gearing moves the stirrer R at the rate of 40 rev. a minute; the latter is made of wrought iron, and is most efficient when the two sets of blades move in opposite directions; when this is not the case, the pan itself should be provided with fixed traverses armed with vertical cross-teeth. In making soap with this apparatus, 1000 lb. oil are run into the pan with the curb O in its place, and heated to 280°–320° F. (138°–160° C.), according to its quality. At this point, 190 lb. of soda-ash for a neutral soap, or 210–225 lb. for a strong soap, dissolved in the proper quantity of water, and at 212° F. (100° C.), is let into the pan at such a speed that it occupies not less than 6 nor more than 12 minutes. The whole is well stirred meanwhile, and swells up enormously; but 5 minutes after the last portions of alkali have been added, the mass subsides, and in 15 minutes more changes from a spongy to a clear, soft, brilliant, homogeneous paste. The curb is then removed, and in about an hour 100 lb. of boiling water is let in from the rose-spout of the soda-feeder, and the whole is again well stirred; if it be desired to mix sodium silicate or anything else with the soap, it is added at this stage, after which, the soap is transferred to the cooling-frames (Chap. VIII.), and a fresh batch is proceeded with. Soap thus made has the following composition:—Oleic acid, 65·0 per cent.; soda, 6·7–7·50; water, 27·50. When rosin is



FIGS. 35-37.
SOAP PAN.

used, it should be added to the oil while the latter is being heated; or the rosin soap may be made in a separate pan, provided with a Morfit's steam-twirl (Fig. 38), in which the tubular blades of the stirrer are perforated so as to emit steam while the whole is in motion; 1200 lb. rosin and

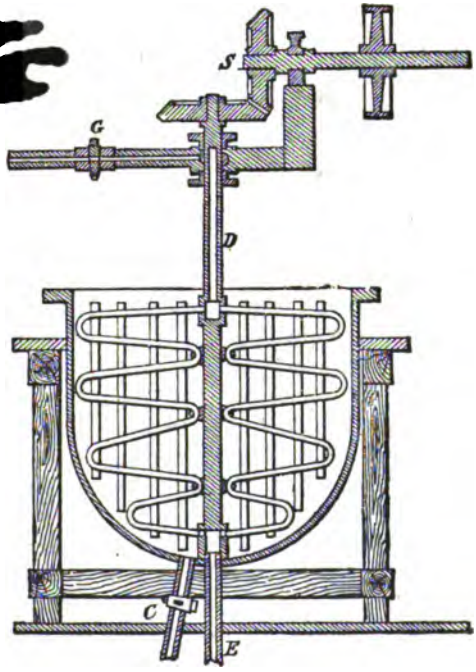


FIG. 38.—MORFIT'S STEAM TWIRL.

2200 lb. caustic lye at 16½° Tw. (11° B.), are boiled together, and the thin jelly so produced is transferred in suitable quantities to other pans. Its percentage composition is:—Rosin, 54·5; soda, 7·8; water, 37·7. The apparatus described here is also suitable for several other kinds of soap, the steam-twirl, &c. being especially useful for making

"hydrated" soaps (p. 166). It is not improbable that such an apparatus as this may be extensively used in the near future, especially if the French process, so largely adopted in America, for deglycerining neutral fats before they are saponified (*vide* Chap. XVI.), comes into general favour.

I*b* (*a*).—The so-called "cold process" consists in mixing given weights of fat, or a mixture of fats, previously melted at as low a temperature as possible [about 109°–111° F. (43°–44° C.)], with caustic soda solution of a given sp. gr. and at about the same temperature, the quantities of each being so adjusted that only just enough soda shall be present to completely saponify the fat. After thorough incorporation, the mixture is covered up, and allowed to stand. In a few hours, the chemical reaction commences, accompanied by considerable evolution of heat, and the soap is formed. After the lapse of 2 or 3 days, it is usually hard enough for use. It is obvious that soaps made in this way retain all the glycerin originally combined with the fatty acids, disseminated through the particles of soap. This, and the comparatively low temperature at which the soap is made, are the chief reasons why this process is much in vogue for the cheaper kinds of toilet soap, since the perfumes employed are not dissipated by heat. It is found, however, that soaps thus prepared are vefy apt to contain an excess of alkali, and hence they are unavailable where perfectly neutral soaps are required. Another objection is, that, as there is no opportunity of removing any extraneous matter, the materials employed must be of the purest, and as the soda lye is usually required in a concentrated (and therefore expensive) form, the process is not so advantageous as at first sight appears. It is chiefly applicable to soaps made on a small scale; when larger quantities are operated

FIG. 39.—HAWES' BOILER.

on, a mechanical agitator, such as Hawes' boiler, represented in Fig. 39, is necessary. This, for operating on 2½ tons of tallow, is a cylinder 6 ft. diam., 12 ft. long, with a central shaft provided with radiating arms, set in rotation by any convenient mechanism. Any saponifiable fat or oil may be used, and for every 100 lb. of the pure fat, 50 lb. of caustic lye at 66½° Tw. (36° B.), should be taken. When this is not very pure, i.e. if it contains much extraneous soda salts, especially sodium chloride, saponification will not take place unless some proportion (10 per cent. on the fat, at least) of coconut oil be used. The following mixtures will be found useful for this process :—

	Tallow.	Lard.	Palm Oil.	Coconut Oil.	Rosin.
(1)	100	..	..	..	..
(2)	100	..	..	50	..
(3)	..	..	55	55	100
(4)	50	30	20	..	..
(5)	..	100	..	..	..

Nos. 1, 2, and 5 make good toilet soaps, which are improved if about ¼ of the soda used is replaced by an equivalent quantity of potash. No. 4 with unbleached palm oil gives a fine yellow soap, liable, however, to bleach in the light. No. 3 is given on the authority of Cristiani, who recommends the use of 100 lb. lye at 42° Tw. (25° B.) to the 210 lb. mixed fat and rosin.

The time required increases with the amount of materials operated on at once. The chief points needing attention are, to use pure materials, to avoid excess of alkali, and so to manage the temperature and the stirring as to make a complete mechanical mixture of the melted fat and lye which will not separate before the chemical combination, i.e. saponification, takes place.

Lombardon secures complete saponification of the fatty matters by means of a compound containing 10 parts soda

subcarbonate, 10 of quicklime, 1 of alum, and 1 of common salt; the whole well boiled with the fat. All the glycerin goes into the soap.

Destibeaux melts together stearic acid and olein (say 56¼ lb. of the former, and 43¾ lb. of the latter) at 140°–167° F. (60°–75° C.) and adds 250 lb. caustic soda at 12° Tw. (8° B.) containing anhydrous soda equal to 11 per cent. of the weight of fatty matters.

Weineck makes soaps without boiling the fats with the lye, thus obtaining a spent lye containing no salt. The fat is melted in a jacketed pan; then about 20 per cent. of a solution of 2 parts soap in 100 of water is added with constant agitation. About 50 per cent. of caustic lye at 71½° Tw. (38° B.) is required for hard neutral soap from tallow. The water in the jacket is heated up to 194° F. (90° C.) till saponification is complete. The spent lye contains only glycerin and "caustic lye."

I*b* (*β*).—This class comprises the soaps which are produced by boiling under pressure. This process has been the subject of numerous patents at various times, all having for their object the shortening of the time occupied in the ordinary methods of open boiling (I*c*), and the saving of the salt employed therein. In this case also, the quantity of alkali employed, whether caustic or carbonate, is accurately adjusted to the fat to be saponified, and the glycerin is retained in the ultimate product; mixtures of any saponifiable fats and resins may be employed. The kind of apparatus used is shown in Fig. 40; it consists of a steam-boiler provided with a manhole and safety-valve, with a feed-pipe A and discharge-pipe C, and with a long thermometer B, in a pocket filled with paraffin. The proper quantities of fat and caustic lye are let in through A; all taps are closed; a fire is kindled, and maintained until the thermometer rises to about 310° F. (154·4° C.), equivalent to a steam pressure of 63 lb. a sq. in. When it has remained at this point for an hour, the tap at C may be opened, and the contents dis-

charged into a cooling-frame D by the steam pressure in the boiler. For a good yellow soap, 7 cwt. tallow, 3 cwt. palm oil, 3 cwt. rosin, and 140–150 gal. caustic soda lye at 34° Tw. (21° B.) are recommended by the inventor, Dunn. Another formula is 800 lb. tallow, 200 lb. palm oil, 400 lb. rosin, 175 gal. caustic soda lye at 42° Tw. (25° B.) for 1 hour at 252° F. (122·2° C.), or 17 lb. steam pressure. It is obvious that this will make a drier soap, since lye at 42° Tw.

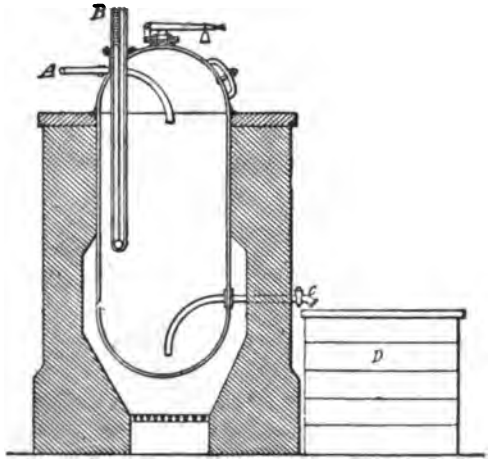


FIG. 40.—PRESSURE BOILER.

contains less water than at 34° Tw., and that the quantity of water desired in this product can thus be regulated to a nicety.

In 1865, Bennett and Gibbs, of Buffalo, New York, took out a patent for effecting this operation with carbonated alkali, thus avoiding the expense of causticising. The boiler is similar to that shown in Fig. 40, but is placed horizontally, and provided with an agitator similar to that for Hawes' boiler. The process requires a higher temperature and pressure than the previous one, ranging from 350° to 404° F.

(176·6°–204·4° C.), or 220–280 lb. a sq. in. The outlet-pipe is provided with a safety-valve, and the inventors state that if this be loaded to about 250 lb. a sq. in., and the raw materials be pumped in at one end, the process may be made continuous, finished soap coming from the outlet, produced in less than one hour from the introduction of the raw materials. The formula recommended is, for every 100 lb. of saponifiable fat, 30–33 lb. of soda ash of 48 per cent. dissolved in 100 lb. water. In the early stages of the process, the liberated carbonic acid is allowed egress by one of the safety-valves, and if any liquid escapes before a temperature of 325° F. (163° C.) is reached, it should be returned to the cylinder. The following advantages over ordinary processes are claimed:—(1) Rapidity of manufacture, (2) improvement in quality, (3) increased yield of soap, (4) economy of labour, (5) saving of fuel, (6) use of cheaper fatty material, (7) saponification of the whole of it, (8) uniform certainty of results, (9) retention of glycerin, and improvement of product thereby, (10) ability to use carbonated alkali. It is obvious, however, that the risk of explosion is not slight, and the practical difficulty of working the agitator at that temperature and pressure must be considerable.

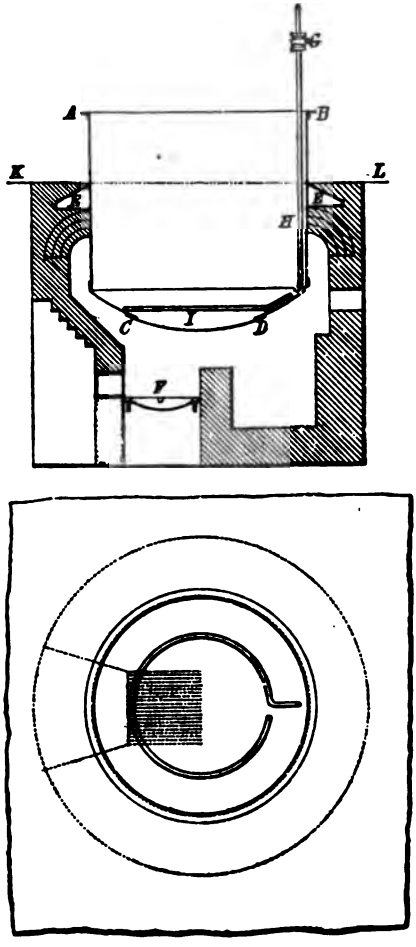
Liebreich aims at securing direct saponification of oils contained in seeds by subjecting the seed to a temperature of 212°–356° F. (100°–180° C.), pulverising, and treating with alkali in excess. The half-stuff thus obtained is watered, and filtered off from the cellulose, &c.

Ic (*a*) *Soft Soaps.*—The production of these is the simplest case of the process most usually adopted in the fabrication of soap, viz. boiling in open vessels, technically termed "coppers," with the aid of steam (either wet or superheated) or of fire, or of the two simultaneously. It will be convenient, therefore, to describe here the construction and fittings of the various kinds of soap-coppers (or soap-pans), and the different modes in which steam and fire are applied to boil their contents.

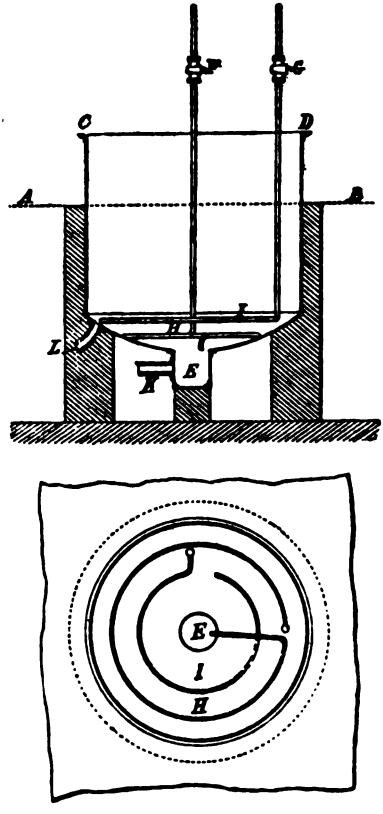
In the days when there was an excise duty upon soap, "coppers" were usually of what is now considered a very small size, and were constructed of cast iron; they consisted of hemispherical pans, upon which were mounted as many cylindrical rings as were necessary to make the copper of a suitable depth, usually about twice its diameter; the rings were joined to each other, and to the hemispherical bottom, with cement joints. There was no limit to their size, except the difficulty of making large castings, and they were usually encased in masonry, and fitted with fireplaces and flues in the manner to be presently described for modern wrought-iron "coppers." In the case of those boiled by fire (the only method until steam-boiling was introduced), the hemispherical bottoms were very apt to crack from overheating, and from many other causes, which it is scarcely necessary to detail, as these pans are fast becoming obsolete.

The removal of the excise duty in England, in 1853, gave an enormous impulse to the soap industry. Manufacturers were no longer deterred from making large batches of soap by the fear that, if they were spoiled, double duty would have to be paid when they were re-made and produced fit for sale; and, as a natural consequence, numerous experiments were tried, both with the raw materials and the apparatus employed. Soap coppers are now made of colossal size, those capable of turning out 50 tons of finished soap (112,000 lb.) at one operation being by no means uncommon, and some of the large American manufacturers have built even still larger coppers, requiring a factory of 3 stories to contain them. Although it is desirable that those boiled by fire should be circular in shape, and not too large —say 20 tons capacity—the coppers whose contents are boiled by steam may be of any desired shape, circular, oval, or rectangular, provided that the steam-pipes be carried into the corners (if any), and be so arranged as to ensure uniformity of ebullition throughout the whole mass. There is no necessary proportion between diameter (or superficial area) and depth; English soapmakers are more accustomed

to pans whose diameters are to their depth as 1 to 1, 1 to 1·25, or 1 to 1·5 (e. g. a pan 15 ft. diam. and 15 ft. deep will turn out 25–30 tons of soap); while their American



FIGS. 41, 42.—FIRE BOILING PAN.



FIGS. 43, 44.—STEAM BOILING PAN.

confrères, less trammelled by tradition, increase the ratio as far as 1 to 2, 1 to 2·5, or even 1 to 3.

Soap-coppers are now almost invariably built of wrought-iron plates, riveted together in the place where the copper is eventually to stand. Figs 41, 42 show a simple form of copper for fire-boiling, with the fire-place, flues, &c.; A B D C

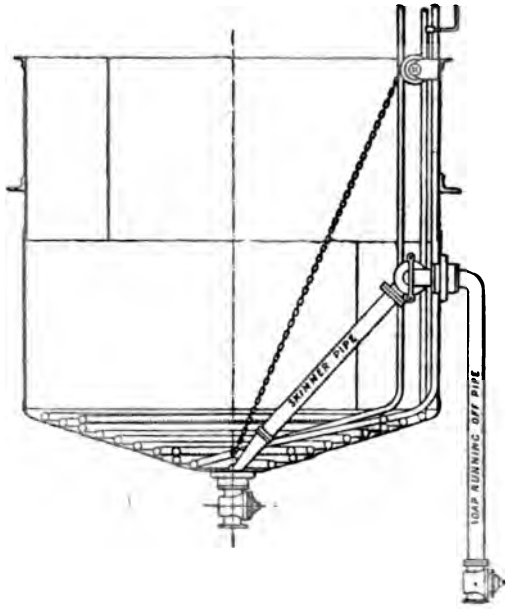


FIG. 45.

is the outline of the copper, C D being a circular renewable plate, in the part most exposed to the action of the fire F. At E are supporting lugs of cast iron; K L is the floor-level; H I a steam pipe ending in a perforated coil, steam being controlled by the cock at G. Figs. 43, 44 show a copper where steam only is used: A B is the floor line; C D E the copper, provided with a "hat" at E to receive

impurities that subside, and to enable spent lye to be removed completely by the draw-off at K. Another draw-off is fitted at L; 2 steam worms are provided: H, with cock F, whose coil is perforated, admitting "open" or "wet" steam among the copper contents, and I, with cock G, in which high pressure or superheated steam is circu-

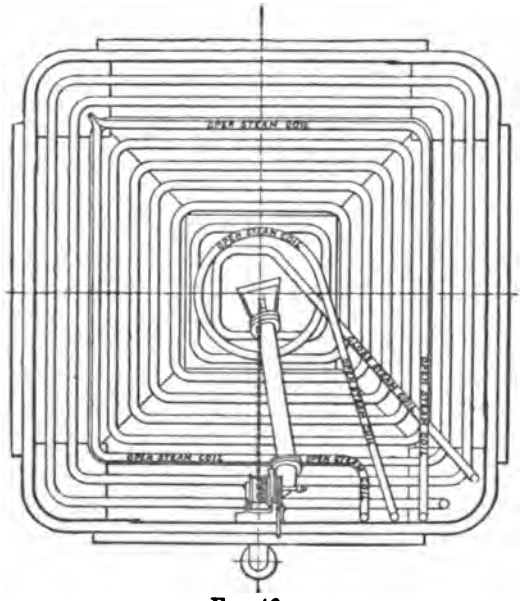


FIG. 46.

lated, for use when it is desired to evaporate water. This last coil is usually omitted in the largest coppers, being only used in making curd and mottled soaps.

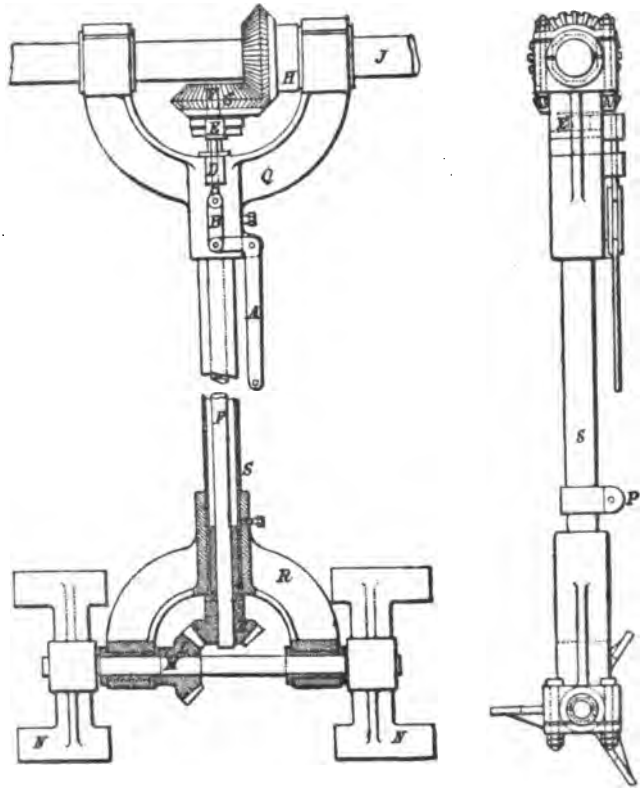
In Figs. 45, 46 is shown one of the newest forms of pan, in which dry or wet steam may be used at will. It consists essentially of a square tank made of riveted steel plates, with a dished bottom and rounded corners, and fitted with dry and wet steam coils and spent lye tap in the bottom. Work-

ing on a swivel joint and adjustable by a chain at any height, is a "skimmer pipe," while the fluid soap is run off by gravity through a down pipe. The apparatus is made by Neill, St. Helens.

An important adjunct to a soap-copper is a little piece of machinery for preventing the contents from boiling over, as they are apt to do when saponification is taking place, and also in a later stage, even after the steam is turned off. It is called a fan, and is represented in Figs. 47, 48; it consists essentially of a rotating paddle, whose blades just touch the top of the boiling mass. The motion is derived from an overhead shaft J, on which is keyed a bevel-wheel H, gearing into a similar wheel G; this latter slides on a feather on the shaft F, being thrown in or out of gear by a fork E, to which is attached a rod C actuated by links B and bellcrank A, in the bottom end of which is an eye for attaching a cord which may be drawn to right or left. The lower end of the fan-shaft drives the overshaft M, on which the fans N are keyed by means of bevel wheels K L. The top and bottom of the fan-shaft are carried by bushes driven in at each end of a piece of stout 2-in. steampipe, and the pipe S is inserted in cast-iron frames Q R. Near the lower end of the pipe is a shackle P, to which a rope or chain is attached for lowering or raising the fan, according to the level of liquid in the copper. The whole swings on the axis of the shaft J.

The fabrication of soft soaps will now be described. Soft soap is a more or less impure solution of potash soap mixed with glycerin in caustic lye, and forming at ordinary temperatures a transparent smeary jelly, containing at times, and especially in cold weather, white grains, which are impure potassium or sodium stearates. The most suitable form of copper for making it is shown in Fig 41. In England, whale, seal and linseed oils are chiefly used, and occasionally a little tallow to produce the grains, or "figging," just described, an appearance which serves no

really useful purpose. On the European continent, hempseed, linseed, camelina, and poppy oils are used, and also rapeseed and train oils, especially in summer, since they



FIGS. 47, 48.—SOAP COPPER PAN.

produce a harder soap. In America, cottonseed oil and oleic acid are often employed. Hempseed oil gives a greenish tint much prized by consumers, which may be imitated by

the addition of a little indigo precipitated by potash from its solution in sulphuric acid.

A very desirable, but not necessary, adjunct to the soap-copper is a set of tanks of iron or wood, whose contents per inch of depth are known, in order that the quantities of oil and lye let into the copper may be regulated. In many large factories the practice is to keep a strict account of the quantity of fatty matter and rosin used, but to control the amount of lye according to the judgment of the soap-boiler. Such gauge and store tanks may be in any convenient place, and pipes or open shutes are carried from them to deliver their contents into the copper; suitable plugs and cocks control the flow of the liquid at the pleasure of the operator.

To make an unsophisticated soft soap, a suitable quantity of oil is run into the copper, not exceeding ¼ its total capacity; at the same time, potash lye at 13½°–16½° Tw. (9°–11° B.) not absolutely caustic, but retaining some potassium carbonate, is let in, and the steam is turned on, or the fire kindled, or both; the fan may also be adjusted at the height beyond which the soap is not to boil, and the whole is carefully watched. If the copper has not boiled until a volume of lye has run in equal to that of the fat, the stream of lye should be stopped, and started again when the ebullition commences. If the oil and lye do not appear to have combined, the fire should be checked, the stream of lye stopped, and gentle steam boiling continued until this is the case. It is very difficult, especially with rape oil, to get the alkali to combine, but when once the process has begun, it goes on with tolerable rapidity, with subsequent additions of lye. From time to time, small samples of the soap are dropped upon a glass plate, and after cooling at a temperature as near 46·4° F. (8° C.) as can be obtained, are carefully examined. The soap is good if it is clear and translucent; a fatty border indicates deficiency of alkali; while if the sample be granular, grey, and lustreless, too much lye has been added, a fault that must be corrected by the addition of more oil,

previously mixed with lye at 3° Tw. (2° B.). Should the sample separate on the glass into soap and clear liquor, the quantity of lye is excessive. If the combination be good, and alkali be deficient, stronger lye (at first 23°–25° Tw. [15°–16° B.] then 38°–42° Tw. [23°–25° B.]) may be cautiously added; a sign of saturation, or rather slight excess of alkali, is the appearance of a striped skin, or lye veins, on the surface of the sample. When it is judged that enough alkali is present, the steam is turned off, and a certain amount of water is evaporated by boiling the copper with fire, during which operation the bubbles get larger, the soap being almost laminated, and they make so much noise in their escape that, in the language of the soap-boiler, "the soap talks."

When the soap is finished, a small sample must not glide or be slippery on the glass, nor must it draw into threads when worked up between the fingers and the thumb; a very small ring should appear in the sample in 12-15 min., indicating the necessary slight excess of potash. The soap is filled into barrels while quite hot, and to promote rapid cooling of the mass, cold soap is often added.

A somewhat shorter method, saving the evaporation in the later stages, has been introduced of late. For every 100 lb. oil, 200 lb. lye at 32½° Tw. (20° B.) is required; when liquid fats are used, this quantity is run in at the commencement of the operation; with solid fats, ½ may be taken, and when thoroughly incorporated, the rest may be added, and the soap boiled as previously described.

If it be desired to make a soft soap in which some of the potash is replaced by soda, the proportions of the two lyes must be accurately adjusted to each other, and to that of the fat used. The process was first worked out by Gentele, and improved by Cristiani, who recommends for the saponification of oil by ⅗ potash and ⅖ soda the following formula:—5000 lb. oil, 2674 lb. potash lye at 32½° Tw. (20° B.), 740 lb. potash lye at 42° Tw. (25° B.), and 2353 lb. soda lye at 32½° Tw. (20° B.). If enough steam be not condensed in

the boiling, water may be added to make the whole weigh 12,500 lb.

To produce a grained soft soap (or "fig"), it is essential to use pure potash lye, and to employ some hard fat, the stearic or palmitic acids of which form potash salts whose crystallisation produces the grains, within somewhat narrow limits of temperature, viz. 48·2° and 59° F. (9°–15° C.). The following fat mixtures will produce it:—(*a*) 55 palm oil, 45 oleic acid; (*b*) 55 palm oil, 15 tallow, 30 linseed oil; 70 palm oil, 30 linseed oil. An artificial grain is sometimes given by clay, starch, &c.*

Two kinds of genuine soft soap occur in commerce, whose compositions are respectively:—

	I. per cent.	II. per cent.
Water	50·5	31·5
Potash	9·5	11·5
Fatty acids	40·0	50·0

The question of admixtures with genuine soft soap, after its fabrication has been completed, is one that demands the serious attention of both manufacturer and consumer. They may be divided into 2 classes: (1) those intended to increase the detersive power; and (2) those added solely to cheapen the product. To the latter belong clay, starch, fecula, glue, and a number of other fraudulent admixtures, whose detection will be dealt with under the head of soap analysis. The first class demands a few explanatory words, and contains chiefly two substances, rosin, and sodium or potassium silicate; the manufacture of the latter, and their use in hard soaps, are described in Chap. IX. For soft soap intended for household and laundry purposes, rosin may be substituted for part of the saponifiable material (to the extent of 10–15 per cent. upon the total oil used) without injury, and, in some cases, with actual benefit; in the same class of soaps,

* A valuable paper on the preparation of soft soaps will be found in Dingler's Polytechn. Journal, 1882, Bd. 244, Heft 1, and an abstract of it in Jour. Soc. Chem. Ind., i. 236.

the addition of potassium or sodium silicate (or carbonate) certainly increases the detersive power, especially where hard water is employed. Most of the soft soaps made, however, are used by woollen manufacturers, for wool washing, fulling, scouring and sizing, and there is no doubt that the best soap for these purposes is a *genuine neutral potash-oil soap.* Experience has shown that the addition of rosin has an undesirable effect upon the fibre, and that the presence of soda in any form is absolutely injurious to it. Wool in its natural state is lubricated by "suint," which contains nearly 50 per cent. potassium carbonate, and scarcely a trace of soda; it is evident therefore that in discarding soda, and using potash, the manufacturer follows the teachings of nature. On the other hand, as has been pointed out by W. J. Menzies, a well-made *neutral* soda soap is preferable for wool-washing, &c., to an impure potash soap containing, as it so often does, excess of caustic potash and carbonate. This is the real ground on which, some years ago, Leroux deprecated the use of potash soaps for such purposes, since, at that time, pure caustic potash, such as is now manufactured in large quantities, was unknown. The use of potassium silicate is injurious, since it attacks the fibre of the wool, and in some cases, by its decomposition, even deposits silica therein, greatly to the detriment of the ultimate fabric. So much injury has been done by the use of unsuitable soaps, that many woollen manufacturers have been driven to make their own, thereby, as they think, ensuring purity. This, however, is also a hazardous proceeding, and it would be really more to their interest to state their exact wants, and to pay a proper and fair price for a soap carefully made with all the appliances and knowledge of a large soap factory, than to run the risk of using a product in which, from want of practice or knowledge, a serious oversight had occurred. The excessive desire for cheapness on the part of purchasers has done more than anything else to depreciate the quality of soft soaps (and of others). Further general remarks on this subject, and upon

the desirability of purchasers buying soaps whose composition is guaranteed by analysis, will be found in Chap. XI.

I *c* (β).—The method of making "hydrated" soaps is very similar to that just described. Fatty matter and (soda) lye are run into the copper, and the whole is boiled together, care being taken to avoid an excess of alkali at first; when saponification has taken place, lye is cautiously added until the soap tastes very faintly of caustic alkali, when the operation is finished, and the soap is ready to be transferred to the frames. Marine soap, for use with sea-water, is made in this way, the fatty matter being entirely coconut oil, and the lye being usually at 32½° Tw. (20° B.). This soap is soluble in weak brine, while other soaps are not. It is difficult to make the saponification begin, but once begun, it proceeds with extraordinary rapidity, the united mass of oil and lye swelling up almost instantaneously to many times its volume. In connection with hydrated soaps, Blake and Maxwell give the following table for the quantity of soda lye necessary for their manufacture:—

100 lb.	tallow	require	3800°	at	16½° Tw. (11° B.).
„	coconut oil	„	4100°	„	25°–32½° Tw. (16°–20° B.).
„	palm oil	„	3200°	„	28½°–36° Tw. (18°–22° B.).
„	lard	„	3400°	„	20° Tw. (13° B.).
„	tallow olein	„	2800°	„	28½°–36° Tw. (18°–22° B.).
„	olive oil	„	3000°	„	25° Tw. (16° B.).

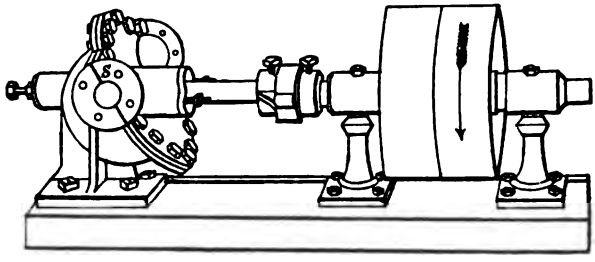
To use this table, divide the larger number of degrees by the degrees Tw., and the quotient is the number of lb. of soda lye at the gravity of the divisor, required to make a hydrated soap with 100 lb. fat.

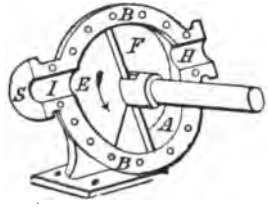
I *c* (γ).—*Hard Soaps*, with soda for a base, made by open-pan boiling, in which the glycerin is eliminated. This class probably includes 90 per cent. of the total soap made in English-speaking countries, and may be divided into 3 kinds, curd, mottled, and yellow. The coppers for their production have already been sufficiently described, but a necessary and hitherto unused adjunct must now be explained, viz. the

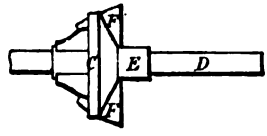
pumps required for changing the lye beneath the soap. They may be placed inside the copper, or outside, and, in this latter case, are connected with the outlet pipes at K L, Fig. 43 (p. 160). For small pans, a simple hand suction-pump answers; for larger ones, a single or double-acting lift or force-pump may be placed inside the copper, and worked by hand, or by an eccentric on a shaft. In large soap factories, some form of centrifugal pump will be found very useful; the usual objection to the use of these pumps, viz. the need of constant lubrication, being obviated by the fact that, so employed, they lubricate themselves. Their great advantages are the absence of valves and of easily deranged working parts, and the large amount of work they will do in a short time. In England, the names of Gwynne and Appold have long been connected with centrifugal pumps; in America, the one most usually employed is Hersey's patent rotary soap-pump (Hersey Bros., Boston, Mass.), which is represented in Figs. 49, 50, and 51. The pump should be placed as little as possible above the outlets in the coppers, and connected therewith by 2½ in. iron pipes, provided with valves. The pipes inside the copper, communicating with the outlets, have swing joints, so that they can be raised or lowered at pleasure. To avoid the pipe-system becoming choked by soap congealing in it, a steampipe should be inserted at one end, to warm the pipes and pump previous to use, and to "blow out" all their contents at the end of the operation. In the figures, S is the suction-pipe; H, the delivery; F, the blades set upon a cone (the rotation of which in the closed case produces the pumping), which is kept in its place by adjustable set-screws. This pump will transfer to any desired part of the factory, lye, melted fat, finished soap (if not too stiff), "nigre," and soft curd. The diameter of the pump is 10 in., of its outlet 2½ in.; when making 120 rev. a minute, it will pump 6000 gal. an hour, its contents being twice emptied in each revolution.

Whatever kind of hard soap is to be made, the first stages of the process are the same for all; but since a curd or a

mottled soap requires the use of fire or "close" steam to evaporate water during the final stages, it is desirable to commence making those in coppers so provided, and either high-pressure or superheated steam may be used in the close-steam worm. Yellow soaps may be made in coppers furnished only with an "open" or "free" steam worm. A useful addition to any copper, giving more room to boil, and hence adding to its capacity, is a "curb" or ring 2–3 ft. high,







FIGS. 49, 50, 51.—HERSEY'S ROTARY SOAP PUMP.

loosely fitted on in segments above the angle iron of the top ring of the copper itself, and capable of easy removal. If, as in Fig. 43, the copper project 2½ ft. above the floor, a "curb" 2½–3 ft. high may be conveniently added, and the fan adjusted so that its blades revolve about 1 ft. below the top of the curb.

To commence a boiling of hard soap, melted fat and caustic soda lye (hereinafter only called lye) at about 16½° Tw. (11° B.) are simultaneously run into the copper, and the steam

is turned on; the same precautions to prevent an excess of lye must be observed as are detailed in making soft soap; if lye stronger than 18° Tw. (12° B.) be used at this early stage, saponification will not take place. When the contents of the copper present the appearance of a homogeneous magma or paste, lye of a higher sp. gr., say up to 42° Tw. (25° B.), may be cautiously added, but it is not essential to do so. The boiling, and the addition of fat and lye, must be continued until a small sample cooled between the fingers has a tolerably firm consistence, and when applied to the tongue, no caustic taste, or only a *very* faint one. Should there be a strong taste (or "touch," to use the American term), or should the sample separate into soap and liquor when squeezed, too much lye has been admitted, and more fat must be added. Should the sample be soft and greasy, more lye is required, especially if any unsaponified fat be visible; occasionally the 2 conditions obtain, both caustic liquor and fat appearing in a sample, which is evidence that combination has not taken place; the remedy is more boiling, with occasionally the addition of water. Practice alone will enable the operator to judge of the completion of this first operation, called "pasting" (French *empâtage*). In English phraseology it is called "killing the goods" or raw material, and the soap is then said to be "close" or in a "hitch" or "glue." In this condition, the soap should contain about $\frac{9}{10}$ of the total soda necessary for complete saponification, with a large excess of water, which is separated from it in the next stage.

Separation (French, *rélargage*).—To effect this, a quantity of common salt is sprinkled into the copper while still boiling, or the strongest brine at 40° Tw. (24° B.) is run in. Since the quantity necessary depends entirely on (1) the sp. gr. of lye used in the last operation, (2) the amount of condensed water where free steam is used for boiling, and (3) whether there is much coconut or palm-kernel oil in the fat employed, no directions can be given as to how much salt should be added; the addition should be made cautiously and

gradually (taking care to allow time for the solution of the salt), and continued until a small sample removed upon a spatula or trowel allows *clear* liquor to run from it. During this operation of "graining," the contents of the copper are very apt to boil unsteadily, and occasionally to boil over with great violence. When this point is reached, the whole process should be stopped, and the steam being turned off, the copper should stand at least 2–3 hours. Its contents then divide themselves into 2 portions, the upper consisting of soap-paste holding about 40 per cent. water, and the lower of a solution known as "spent lye," containing common salt, any carbonate and other sodium salts present as impurities in the original lye, and nearly all the glycerine of the fat employed. It should contain *no caustic soda*, and *no soap* or saponifiable material; if it contains the latter, enough salt has not been used. For the presence of caustic soda, a sensitive tongue will be found a sufficiently delicate test, while the addition of a mineral acid will throw up a scum of fatty matter, if any be present; it will also be found useful to observe the sp. gr. of the spent lye, as a means of controlling the amount of salt used. After the copper has stood for some hours, the spent lye should be pumped off, and, if there is then sufficient room, more fat may be run in, and the whole operation repeated: at this stage, the rosin is usually added for a yellow soap, being broken into lumps and shovelled in, unless it is combined with soda in a separate copper, and mixed with the fat-soap in the next operation.

Clear-boiling (French, *coction*).—All the goods having been "killed," and the spent lye removed, a small charge of lye at about 18°–21½° Tw. (12°–14° B.) is run in, and the copper boiled for 2–3 hours; at the end of this operation, the soap should have a faint but decided caustic taste, and a small sample on a spatula should allow clear lye to run off it; if this be not the case, more, and in some cases stronger lye, must be added. This operation communicates additional soda to the soap, and washes out, as it were, some of the salt entangled in it. After some hours' subsidence, the "half-

spent" lye that sinks to the bottom is pumped off, and may be used in another copper for "killing" more fresh goods; the soap from such lye, however, will be of an inferior colour.

The next stage of this operation is to boil the copper with open steam; if the contents are not perfectly homogeneous, and in a state resembling a stiff paste, i. e. if the copper be not "close," but have a tendency to separate into lye and soap, when examined on a spatula, the sp. gr. of the entangled lye is reduced by the addition of water, until the desired condition is reached. A small stream of lye at about 18° Tw. (12° B.) is then allowed to trickle in, until the homogeneous paste again separates into flakes of soap and clear lye, the boiling being continued all the time; the soap should now taste strongly of caustic soda, and feel hard when cold; this operation is technically called "making" the soap, and when enough lye has been run in, boiling should be continued for some hours, to ensure complete saponification, since it is very difficult to make neutral fats take up the last portions of soda. The large coppers previously alluded to require a whole working day (12 hours) for this operation.

The operations above described, may *in experienced hands* be somewhat reduced in number and time, but much greater care is then required. By the proper use of lye of various degrees of concentration, and of salt, it is possible to "kill" 40–50 tons of mixed tallow and rosin in one copper in a day—to dispense with the next operation—and to "make" the copper on the day following, finishing it on the third day. The mode of finishing depends entirely on the kind of soap required.

Curd Soaps.—The raw materials for curd soaps should contain no rosin, and but little, if any, coconut or palm-kernel oil, but any other oils or fats may be used. White curd is usually made from tallow or lard, or a mixture thereof; brown curd, from bleached palm oil, or kitchen grease, or bone tallow; and manufacturers' curd soaps, from various fats. When the soap is "made," the open steam is

shut off, and the boiling is continued either with fire or with close steam; this concentrates the lye, and the flakes of soap gradually approach the spherical form. From time to time the boiling is stopped, the sp. gr. of the lye is observed, and a sample of the soap, from which the lye has been allowed to separate, is put out to cool. When it is sufficiently hard, the boiling is finally stopped, and after a few hours' subsidence the soap is ready to be removed; the amount of water left in it varies inversely as the sp. gr. of the lye in which it is boiled.

Curd soaps not only take first rank among bar soaps, but of late years they have attained the largest consumption. Laundry curd soap is much used in Germany, and where clean grease and caustic soda are available, it is generally made by the direct process. The greases which find use in its preparation, besides coconut and palm-kernel oils, are cotton, peanut, palm and bleached linseed oils, elaidin, bone grease, animal grease and lard mixed together in varying proportions.

As late writers on the subject have proposed the use of such grease as each thought would yield the greatest amount of soap, Eichbaum has recently been working to the same end with the so-called "kammfett," the animal grease mentioned above which is composed of a mixture of tallow, lard, and the grease from horse fat, and with firm white olein (obtained by distillation from waste mill grease) in combination with palm-kernel oil.

He has tried this white, firm olein by direct boiling in various combinations, and has obtained very satisfactory results. For instance, he proceeded with a batch consisting of 1584 lb. of palm-kernel oil, 220 lb. of animal grease, and 1276 lb. of white olein as follows:

The scraps at hand, together with about the same quantity of caustic soda lye of 44° Tw. (26° B.) as there will be of grease, are placed in the kettle and melted. The animal grease is then added and boiled in, and the liquid palm-kernel is crutched in, in divided portions. After about 52 lb. of

salt are added to the soapy mass, the firm olein is added gradually. About 8 lb. of soda crystals should now be thrown into the pasty mass, the whole boiled free of foam and strong salt water added with constant stirring, until the paste feels wet, and the grains flatten out on bubbling up. The operation of separation should be conducted very carefully, as otherwise the soap will be entirely salted out. If it should happen that the soap has been too much salted, and the lye drops from the paddle when taken out, the soap should again be drawn together by adding hot water, until a smooth paste is obtained that is readily moistened.

The kettle should now be covered for a few hours, and then, when the handsome, honey-yellow soap is removed that results from carrying out the process correctly, there will be found a thin layer of dark paste containing all the impurities present, below which is the spent lye. By this method, but little of this dark paste is formed, but about 145 per cent. of pure white soap is obtained.

When cooled in the forms, this soap exhibits a silvery lustre, and is quite firm. After long drying it generally assumes a light yellowish colour, and becomes somewhat transparent, which is rather an advantage.

Mottled Soaps.—The term is here used to denote the old-fashioned curd-mottled soaps, not those marbled with blue, grey, or red, which have appeared in the English and foreign markets within the last 30 years, and which will be described in Chap. IX. In the fabrication of soap, it is impossible to avoid entirely the presence of earths and metallic oxides. These consequently decompose a small portion of the soap, combining with its fatty acids, and forming soaps of lime, magnesia, and iron (from the "cappers"), which though insoluble, are softened by heat, and disseminated in a state of minute division through the soap paste: any slight impurities in the fat employed, when not dissolved in the caustic soda solution, are similarly diffused. If, after a soap is "made," the lye in which it is suspended is concentrated to a point

short of that necessary to produce hard curd soap, and it is then transferred to the cooling-frames, with a certain quantity of lye entangled in it, these insoluble particles will, during the solidification of the soap, collect together, and produce the appearance known as "mottling"; and the effect is heightened by the partial crystallisation of the soap. No rule can be given for the point of concentration; it varies with the fat used, with the amount of lye in the copper, with the quantity of salts other than caustic soda in them; and in short, the proper "mottling condition" is a physical one, chemical tests being of little use in deciding it. Nothing but practice and careful observation can make a successful mottled-soap boiler. The principle of the process has been clearly laid down; and the various formulæ given in books, involving in many instances several changes of lye, are but different modes of arriving at the same result, viz. the suspension of pure soap, and of soaps of the metallic oxides, in soda lye of a given sp. gr. If the soap be boiled too long, it "sets" in cooling before the mottling has had time to aggregate; if it is not boiled enough, an undue quantity of lye remains in the soap; but from their mode of manufacture, mottled soaps must always contain some lye in the cavities between the curds; hence they are the most suitable and really economical soaps for washing clothes, &c., in hard waters, although not, of course, for toilet purposes. It not unfrequently happens that the soap-copper does not contain enough metallic soaps, &c., to produce a definite mottle. In this case, some "mottling" is added; for a grey, Frankfort-black, or very finely levigated manganese black oxide, may be used; the peculiar greenish mottle which becomes red on exposure, characteristic of Marseilles and Castile soaps, is produced by adding some solution of iron protosulphate to the copper when the soap is nearly finished (about 4 oz. of the salt to 100 lb. fat); the precipitated iron protoxide suspended in the soap is greenish, but it becomes peroxide in contact with air, to which the change to a red colour on exposure is due.

In England, mottled soaps are usually made from kitchen grease, and from bleached palm oil. In Marseilles, mixtures of various seed oils are used, of which olive oil is the principal, and cotton-seed, poppy, hempseed, gingelly, and ground-nut oils are frequent components. In these mottled soaps, little or no coconut or palm kernel oil should be used, although such oils form an almost essential constituent of the new mottled soaps referred to above.

Yellow Soaps.—The finest yellow soaps are made from the best tallow and rosin, which last is an essential constituent of them; in some cases, lard, or lard stearin is used. Inferior qualities may be made from the "nigres" of better sorts, from bleached palm oil, greases of all kinds, and in fact from any saponifiable solid fat; fluid oils must be used, if at all, in small quantities and with caution. The proportion of rosin may vary from $\frac{1}{8}$ of the total fat, to an equal weight, or even more, according to the quality of soap required. In the south and west of England, the very best quality is known in the trade as "primrose," and is made from the finest (unbleached) tallow and "window-glass" rosin; the lowest grade of brown is made from the "nigres" of the grades above mixed with curriers' grease, leather tallow ("sod-oils"), and other dark and foul but hard fats, with black rosin.

Although rosin is insoluble in water, its compound with soda is soluble, the solution frothing strongly like that of the fatty soaps, which, in most respects, it closely resembles. The chief point of difference is the granular character of the solid compound, which prevents its being compacted into masses having the sectile consistency of the fatty soaps. But it is nevertheless capable of being incorporated in considerable proportion with the fatty soaps, without sensibly affecting the consistency of the product, although the colour and smell of the latter are thereby modified; in fact, the characteristic colour and odour of "yellow soap" are due

mainly to the rosin in it.* It is scarcely necessary to point out that no question of adulteration can be raised in this connection. The soda-rosin-acid salts are valuable detergents, whose application alone would be very limited, owing to their inferiority, in certain points, to the soda-fatty-acid salts. But as this is duly recognised in the price of the soap, there can be no suggestion of fraud.

The finishing operation for yellow soaps is termed "fitting" in England, and *liquidation* in France, and requires considerable judgment on the part of the operator. After being thoroughly well "made," the copper stands at rest for at least 12 hours; the half-spent lye is then pumped off, and the open steam is turned on. When the copper is again boiling, it should be continued so until its contents are perfectly homogeneous (the time depending much on the size of the copper), and the soap should then be examined with a clean trowel. When in proper condition, a thin layer should drop off a *hot* trowel held edgeways in 2 or 3 flakes, leaving the metallic surface quite clean; but if, as is more probable, the layer breaks up into several small flakes, and the soap is stiff, water should be cautiously added, to reduce the sp. gr. of the still entangled lye. If, on the other hand, the film will not leave the trowel at all, a small quantity of strong lye, at say 23°–32½° Tw. (15°–20° B.), or of brine, may be cautiously added, to cause it to do so. In the first case, the "fit" is said to be "open" or "coarse"; and in the second, to be "close" or "fine." Here again, practice and observation alone enable the operator to obtain "a good fit," and when it is obtained, the steam is turned off, and the whole is allowed to stand. The copper is then covered up with planks, or an iron cover, and kept as warm as possible; small coppers may stand a day or two, large ones as long as a week.

* The odour is due mainly to American rosin, which differs very markedly in this respect from French. Moreover, with grades of equal colour, American rosin gives a soap decidedly superior in this respect to that from French rosin.

During this period, the contents arrange themselves in three layers, (1) a light crust full of air-bubbles, technically called "fob," (2) the finished or "neat" soap, forming about $\frac{4}{5}$ of the whole, (3) the "nigre" which is an impure solution of soap in lye, and contains all the impurities present in the copper. The amount of this last depends entirely upon the character of the "fit." A fine fit gives a very large nigre, containing much soap; while a coarse fit gives a small nigre, composed chiefly of impure lye. The English practice is to fit rather "fine," competition among the various makers for purity and colour being excessive; while the Americans are usually content with a coarse fit.

According to Tardain a good soap can be made from impure fat in the following manner. The fatty matter is boiled and agitated with double the quantity of water and 12 per cent. of slaked lime in a flat-bottomed iron boiler, shaped like a truncated cone. This gives a hard lime soap and a solution of glycerine, the latter being drawn off. The lime soap is slightly washed, and to it is added a solution of sodium carbonate in excess of the lime used. On mixing and boiling, the lime soap is decomposed, lime carbonate falls to the bottom, and flakes of soda soap float, especially if some sea-salt has been added.

Payn treats the fatty matter with lime and water under pressure in an autoclave; after decomposition, the aqueous solution of glycerine is drawn off, and the lime soap formed is decomposed by soda or potash hydrate, precipitating the insoluble lime, and leaving a soda or potash soap, which may be finished in the soap copper as usual.

Cottonseed Oil Soap.—Cottonseed oil is of growing importance in the soap trade, and the following account, from the *American Soap Journal*, of how it is employed by a practised soapmaker, contains much information of a useful nature. The question of how much soap a given quantity of tallow will make is often a difficult point to gauge. In the following method one of the advantages claimed is that the yield

of soap agrees with calculated yield, 181 lb. of fat giving 535 lb. of clear waxy soap. The formula given below has been successfully used for 18 months.

	lb.		lb.
Refined cottonseed oil	164	Starch	3¾
Tallow	16	Sal soda	5
Rosin "K"	75	Silex	40
Silicate of soda "N"	75	Water	1¼
Palm oil	1	Perfume	1
Caustic lye 64° Tw. (35° B.) ..	153		535

The method adopted for working up this formula is as follows:—Commencing with the cooling frame, the materials are framed, thus avoiding framing the soap. This necessitates the use of a good tight frame in which the cottonseed oil, palm oil, and tallow, carefully weighed or measured, according to the proportions given above, are placed, having first been warmed to a temperature of 115° F. (46° C.). The rosin previously warmed and cut with a small amount of weak lye to keep it fluid, is then added.

The frame is now rolled under the mixing machine, which has moveable shafts and blades. These mixer shafts are lowered into the foregoing mixture and set in motion. The caustic lye and the sodium silicate are now added, and the mixture is stirred for 7–10 minutes, when the starch, sal soda, and silex are added, and the whole is stirred for another 4–5 minutes. The mixture should then have a glossy and smooth appearance, indicating that the incorporation is complete. The perfume is added, the mixing blades are removed, and the frame is set aside to cool. The soap will be quite hard in a reasonable length of time. It may be stripped on the second day, and cut up on the third. The difference in cost between tallow and cottonseed oil introduces the element of economy; there is no loss save the moisture which evaporates during the 3 days' cooling; no spent lye; and no nigres. Lastly the soap is found to do good work in actual use in the wash tub, containing less moisture than there usually is in boiled and settled soaps.

CHAPTER VIII.

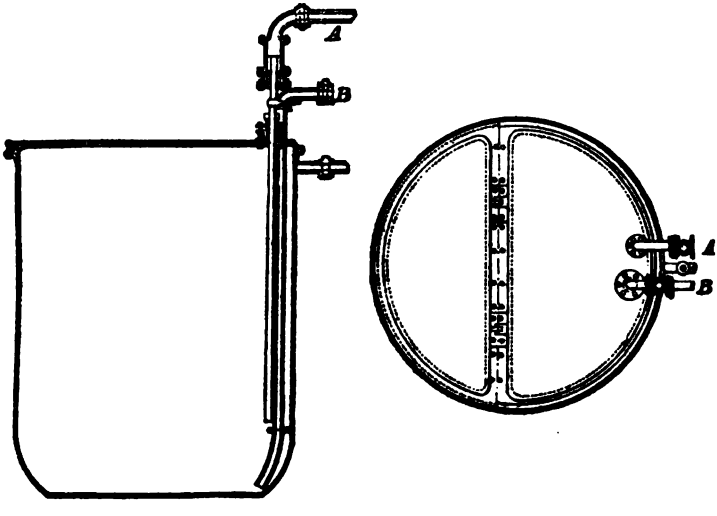
TREATMENT OF SOAP AFTER ITS REMOVAL FROM THE SOAP-COPPER,—Cooling, Cutting, Drying, Moulding.

After the soap has been finished in the copper, it may either be put in the "neat" state direct into the cooling-boxes or "frames," or it may be transferred to the mixing-tanks where various solutions or substances are incorporated with it, prior to its being allowed to solidify. Curd soaps, and most old-fashioned mottled soaps (unless they are so thin, and their curd so flat, that they may be treated like a fitted soap), should always be carefully skimmed off the subjacent lye by ladles, a process which demands very careful attention in the case of curd soaps, lest any portions of lye should be accidentally entangled in the soap, producing want of homogeneity, called "rowiness," seen when the soap is cut up for sale. The same appearance is presented in curd soaps which are framed before they have been boiled dry enough, i. e. when the sp. gr. of the lye and the state of aggregation of the soap particles are insufficient to cause a complete separation, after standing, between the lye and the soap. Mottled soaps, however, not being boiled so dry, i. e. the lye not being so concentrated, always should contain a full proportion of lye, thus mechanically mixed with the curd.

In large factories, fitted soaps are invariably transferred to the frames by suitable pumping machinery. A peculiar method of emptying coppers which contain perfectly homogenous soaps, without any nigre or lye beneath them, was invented by Gossage, and is represented in Figs. 52, 53. An air-tight cover is screwed on to the copper, and a blast of air is turned in through A; the pressure thus exerted forces

the soap out through the delivery-pipe B in a continuous stream, until the lower end of that pipe becomes uncovered, when air rushes through it. This is chiefly used for hydrated soaps, and for the "blue-mottled" soaps, described on pp. 169 and 201.

Soap frames are of two kinds, according as it is desired to cool the soap slowly or quickly. In England, the internal measure of both is 45 in. × 15 in., a relic of the days when a



FIGS. 52, 53.—EMPTYING COPPERS.

duty (removed in 1853) was levied; the width of this makes the length of the English bar of hard soap, which is usually either 2½ or 3 lb. in weight, according to its thickness. Where slow cooling is required, as is always the case with mottled soap, wooden frames, usually made of pine, are employed; and sometimes they are enveloped in a padded covering to conserve the heat, or are hollow (jacketed) so as to admit water of the desired temperature. Their general appearance is shown in Fig. 54; each section or "lift" (2) is

lined with thin sheet iron, the wood being 2½–3 in. thick, and the several sections, each about 9–12 in. deep, should fit closely upon each other when piled in a series (1). The

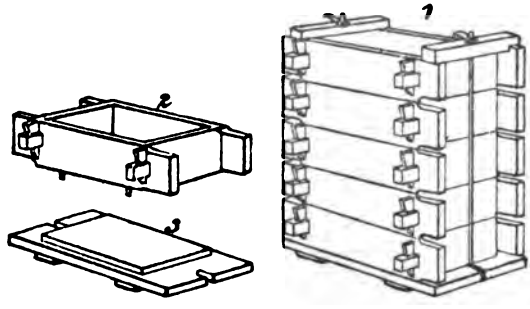
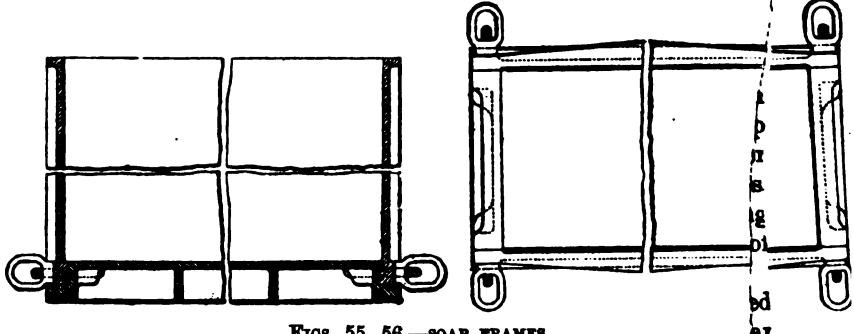


FIG. 54.—SOAP FRAMES.

bottom of the frame (3) may be made of wood or of brick; in the case of curd-mottled soaps, it is convenient to have a well in the bottom, to receive the lye which drains from the soap,



FIGS. 55, 56.—SOAP FRAMES.

especially if, as is frequently the case, the frame is 20–30 ft. high. Most curd and all yellow soaps are cooled rapidly in cast-iron frames of any desired shape and size. Figs. 55, 56 show a longitudinal section and plan of a form frequently

adopted in England, which is almost watertight; the superficial measure is 45 in. × 15 in., and the height 50–60 in. The four sides are held together by bolts and nuts, and when the soap is cold (i. e. after the lapse of 3–7 days for this size), these are unscrewed, the sides are removed, and a solid block of soap is left standing on the bottom of the frame. This

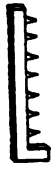


Fig. 57.

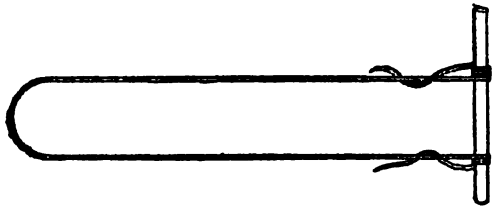


Fig. 58.

may be at once cut up into slabs and bars, or may be slid away bodily to store. Occasionally such frames are mounted upon wheels, for convenience of transport about the factory. During the cooling of stiff soaps, it is desirable, in order to avoid waste in the subsequent cutting, to follow up the contraction of the soap in the frame by slight pressure, exerted either by a screw-top to the frame, or by beating down the soap with a mallet.

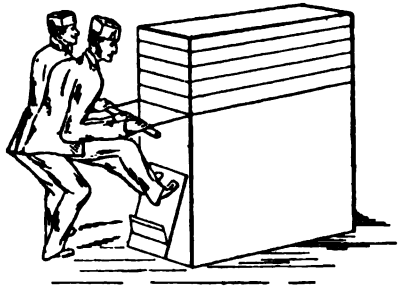
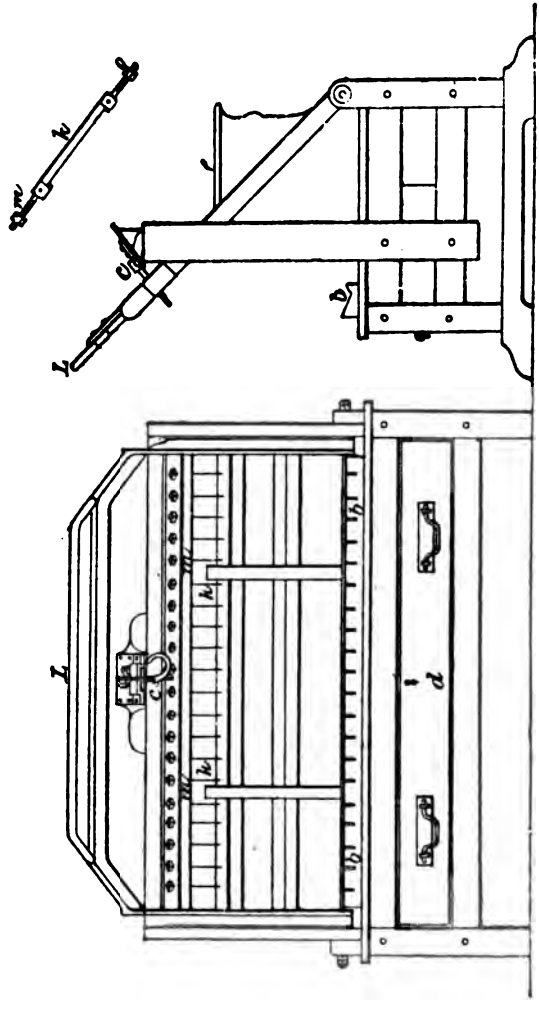


Fig. 59.

When it is desired to cut the soap, the sides of the block are marked with a scribe, Fig. 57, the teeth of which are set at the thickness desired for the bar of soap. A brass or steel wire, Fig. 58, is then taken by two men, and drawn through the block, Fig. 59, which is thus divided into slabs; these



FIGS. 60–63.—SOAP-CUTTING MACHINE.

are at once removed to a machine which will divide them into bars. The cutting machine usually employed in England is shown in Figs. 60–63. The cutter itself is worked by a lever-frame L, which contains wires or for very hard soaps, thin steel knives *k*; the slab is placed longitudinally and nearly upright on the base-board *b*, and the lever-frame is then drawn through it. The bars thus formed fall back upon the shelf *f* behind, whence they may be removed and set aside to get cold. Before repeating the operation, the lever-frame must be raised, and hitched in its place by the spring-catch *c*. The bars when removed from the machine, are piled across each other in "open pile," in such a way that air freely circulates among them. When thoroughly set, they are stored away in "close pile," or packed in boxes for distribution.

In America, Ralston's cutter and spreader, Fig. 64, is largely used; it has an arrangement A B for spreading and stamping the bars, and is very useful where soap is rather soft when freshly cut. The slab is laid upon C, and the cutting-wires are shown at D. Van Haagen, of Cincinnati, has devised a machine for dividing a frame of soap into bars all at one operation.

Röber of Dresden has recently introduced an improved machine, as shown in Figs. 65, 66. It consists of two tables, the smaller of which is moveable and carries the cutting frame. The larger table is fitted with the press plate which forces forward the soap to be cut. The cutting wires are fixed in such a manner to spiral springs that they cannot break even with the hardest soaps, and slipping is avoided. The machine can cut about 15 cwt. an hour with facility. The adjustment of the universal frame for various sizes occupies only a few minutes.

The main objection to cutting machines is the tendency of slabs and bars of soap, when fresh, to adhere strongly together, unless they are immediately separated.

In connection with the cutting-up of soap, it may be conveniently mentioned here that certain soaps undergo a

kind of case-hardening process as soon as they have been barred-up. Most of the French mottled soaps are soaked, or even stored, in weak lye, or weak brine, or a mixture of both;

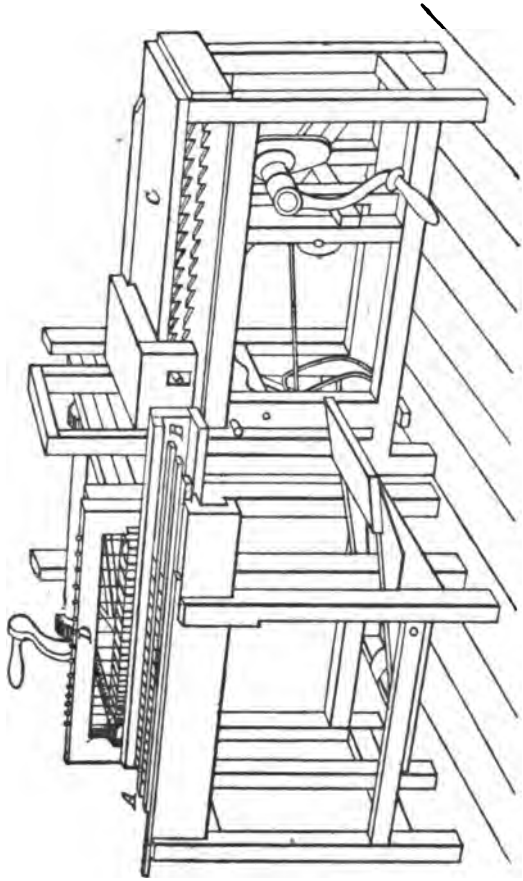
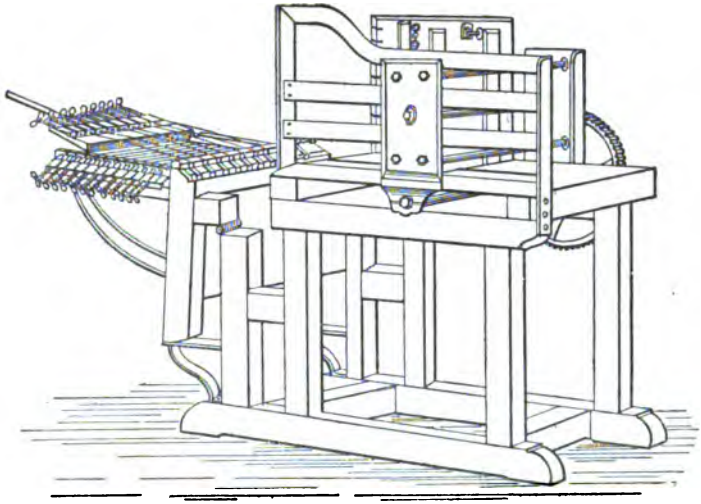
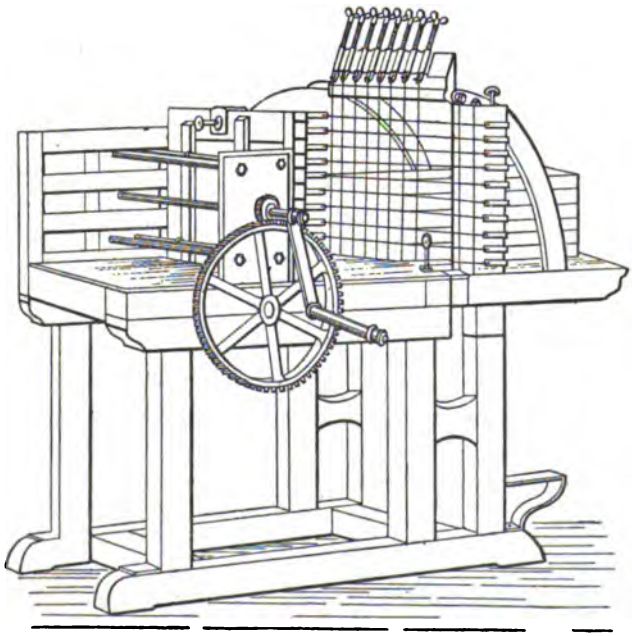


FIG. 64.—RALSTON'S SOAP-CUTTER AND SPREADER.

and some of the English blue-mottled soaps are also soaked in brine. The usual process, however, is a drying one, which may be carried out either by directing a current of warm,





FIGS. 65, 66.—HÖBER'S SOAP-CUTTING FRAME.

dry air, by a fan or otherwise, against a pile of bars, or by spreading the bars in a drying chamber, Fig. 67, which is heated by fire to a temperature short of that at which the soap begins to melt. The fire is kindled in A, and the heated products of combustion pass along E to F, while the air, which enters at H, heated by them rises through the vent-holes O, and, after taking up much moisture from the soap M, passes out through K.

The bars of soap, when freshly cut and still soft, are usually impressed with some words indicating the name or

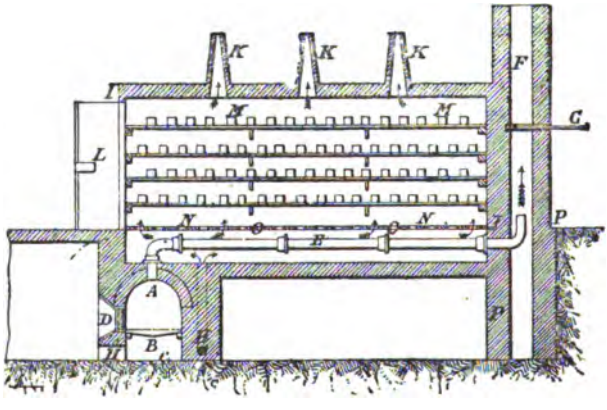
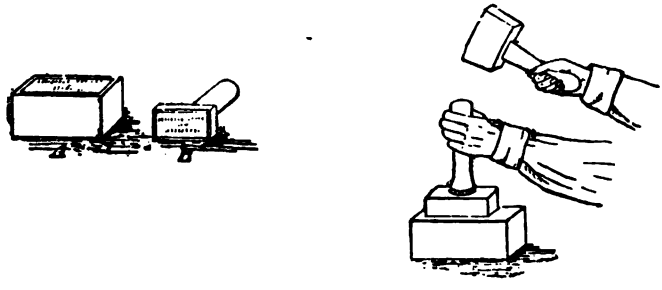


FIG. 67.—SOAP DRYING CHAMBER.

quality of the soap, and the trade mark or name of the manufacturer. This is most simply done by a hand-stamp, in which the letters or device are cut in hard wood or cast in brass (B); the arrangement and mode of using it with very hard soaps are shown in Figs. 68, 69.

In England, it has long been customary to sell soap in bars 15 in. long, weighing 2½–3 lb.; but during the last few years a great demand has sprung up among the retailers for ordinary household soaps cut and stamped into 1-lb., ½-lb., and ¼-lb. blocks, a form which also obtains to a very

large extent in America. Various parts of each country differ considerably in the shapes preferred for these blocks, and the formation of each kind demands a special set of



FIGS. 68, 69.—SOAP STAMPS.

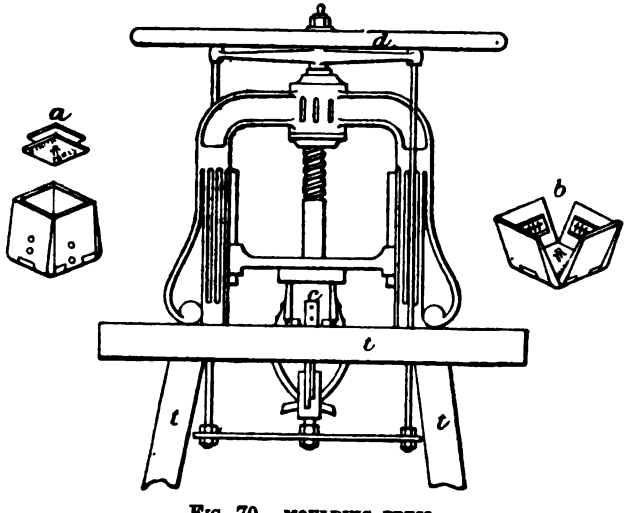


FIG. 70.—MOULDING PRESS.

cutting-wires, and of moulds and dies, for their production. The 1-lb. and ½ lb. blocks are often "semi-cut," so that they can be readily divided into two ½ lb. and ¼ lb. pieces respec-

tively. The simplest moulds are usually cast in brass, each tablet requiring two, producing an upper and an under surface; but occasionally a mould-box *a b* with hinged sides is employed, with a screw-press, such as is represented in Fig. 70. With the ordinary tablets, it is necessary to slightly dry them superficially, and to give them a *very* thin coating of oil that they may not stick to the die. The simplest form of hand press will stamp upwards of 600 ¼-lb. pieces an hour. For larger tablets, a foot-power press is desirable, such as that made by W. H. King, Philadelphia. All large manufacturers, however, employ some form of steam-power press; one made by Neill & Sons, St. Helens, Lancashire, England, is shown in Fig. 71. By moving the handle A, steam is admitted into the bottom of the steam-cylinder D; the piston being forced up, the cylinder lowers the die E into the die-box F. The rod attached to the lever at B works in connection with a die that is always in the die-box and attached to the spindle C, having a slot for the lever to work in such a manner that when the piston is at the bottom of the steam cylinder the bottom die is at the top of the die-box, and when the piston is at the top of the cylinder the bottom die is at the bottom of the die-box; thus the stamped tablet, being raised out of the die-box at each stroke, can readily be removed. The great advantage of the lever working the steam valve is, that the attendant must take his hand from the dies before the blow is given, thus preventing accidents arising through the blow being given when the hands are at the dies. A very ingenious press was patented by F. J. Cleaver in 1870, and

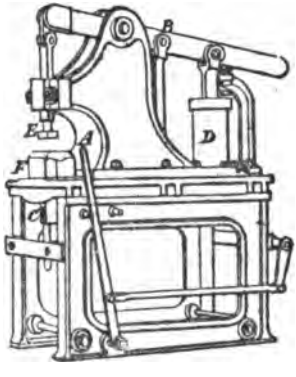


FIG. 71.—STEAM PRESS.

exhibited in action by his firm at the International Health Exhibition, 1884. It comprised a turn-table holding four lower moulds, a pile-driving stamp, worked by a double cam, and an ingenious mechanical finger to pick up the stamped tablets, and deposit them upon a moving belt. Another form (Fig. 72)

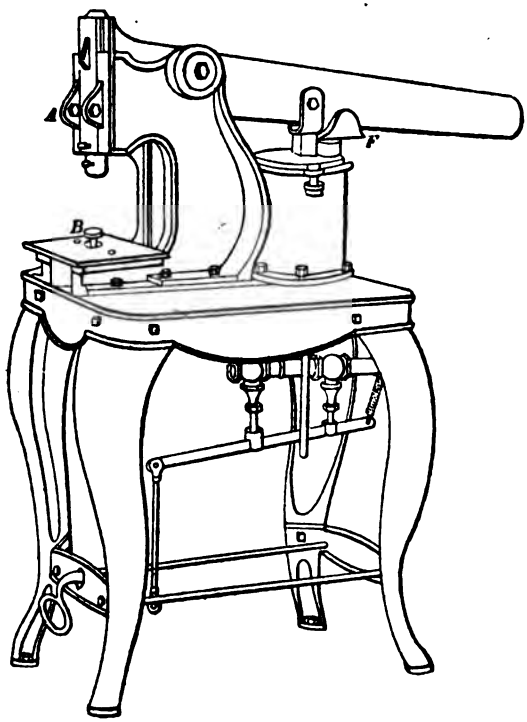


FIG. 72.—STEAM PRESS.

is made by Hersey Bros., Boston, and with it a smart workman can mould 2000 cakes an hour; it is supplied with steam at 20 lb. pressure through a tin pipe.

For moulding transparent tablets of toilet soap, important advantages are claimed for the apparatus made by Cowles

under R. Finlay's patent (No. 4581 of 1893), not only in the direction of economy of labour and material but also as regards variety of effect.

Soap leaves were at one time in vogue for personal use, being put up in little "books," from which a leaf could be taken as wanted. They appealed specially to travellers, as they avoided the carrying of a wet cake of soap in the baggage. Their preparation was described by Buczkowski as follows: Into a mixture of 10 parts glycerin and 35 parts spirit in a boiler, are introduced 60 parts dried shreds of glycerin soap and 50 parts coconut oil soap; the whole is treated at 161½°–179½° F. (72°–82° C.) with constant stirring till reduced to a homogeneous and semi-liquid mass. To facilitate solidification, ½–1 per cent. of oil of turpentine may be added. The liquid mass is poured into a jacketed trough, where the temperature is maintained. In this condition, an endless roll of absorbent material, such as paper or textile fabric, is passed through, excess being removed by rollers.

CHAPTER IX.

SOAP,—Filling and Sophisticating.

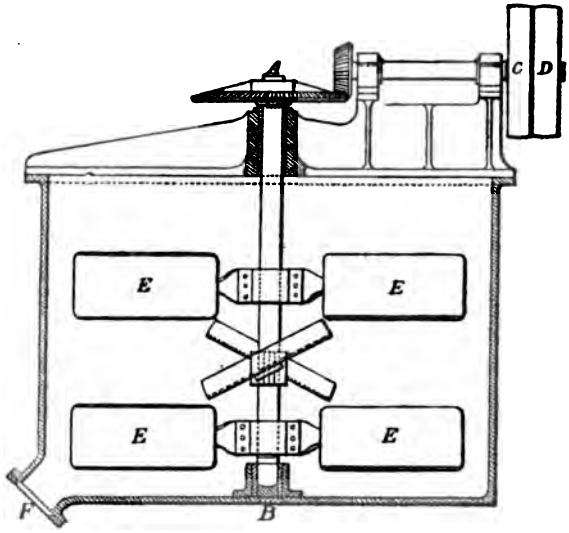
So far, attention has been confined to the fabrication of genuine unsophisticated "neat" soaps, containing not more than 32 per cent. of water when freshly made.

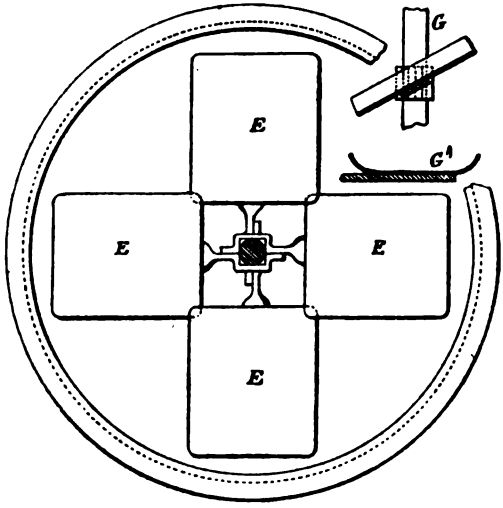
It now remains to deal with the various substances which are mixed, by almost every manufacturer, with soaps after they have been removed from the copper, and the mode of their incorporation, known in the trade by the suggestive name of "filling." These may be classed under two heads: The first class, which will be considered somewhat in detail, comprises all those soluble alkaline salts, such as silicates and carbonates, added to soap to increase its detergent power; between the two classes may be placed water, which is always present to a greater or less extent in filled soaps, and simply reduces their actual value and economical use; while the second class includes *all insoluble substances*, such as clay, steatite (i.e. soapstone, or magnesium silicate), powdered talc, barium sulphate, starch, fecula, as well as all soluble substances like glue and gelatin, which have *no detergent power* in themselves, and are simply added to increase the quantity of water in soaps, or as mere adulterants or make-weights. (A notable example of this is the use of clay, or steatite, 5 or even 10 per cent. of which may be mixed with soap without its presence being apparent to the eye.) For obvious reasons, only the use of the first class will be described in the present chapter; but further remarks on the subject, and methods for detecting and determining the quantity of these adulterants, will be given in Chapter XI. on the Analysis of Soap.

With the exception of the silicated mottled soaps (blue, grey, and red), a special description of which is given on p. 200, all "filled" soaps are made by incorporating the soap-paste fresh from the copper with the "filling," at a temperature of about 170° F. (77° C.). On a small scale, this may be readily done by stirring the two together in the soap-frame with a "crutch," which is a perforated piece of wood or iron, whose flat side is attached at right angles to a pole, by which it is moved by a man vertically up and down in the frame. When many tons have to be mixed, however, machinery in some form must be employed, and the choice of the form thereof depends upon the probable consistency of the mixture. Whatever form be decided upon, it is quite essential that it should not merely mix the soap in one plane, but that the contents of various planes should be intermingled; simple rotation of arms at right angles to a vertical shaft is therefore insufficient.

A suitable arrangement is shown in Figs. 73, 74. The blades E of the various mixers are set at an angle of 45° on the shaft A B, at the top of which is a pair of bevel-wheels with fast and loose pulleys C D; F is the discharge-hole, provided with a valve for drawing off the stiff soap. At G G′, are portions of the mixers and scrapers in section. It is desirable, but not necessary, that there should be some means of controlling the temperature of the tanks or vessels in which the "crutching" (as this mixing process is technically called) is carried on. Close steam-worms or steam-jackets are very suitable for this purpose; they should in all cases, however, be cased with a non-conductor, to prevent loss of heat by radiation.

Where very stiff soap has to be crutched, probably the best arrangement is an Archimedean screw, which is largely used in America, where most of the soap is made quite stiff; this lifts up the various layers effectually, and is most conveniently set inside a jacketed cylinder, whose height is about 1½ times its diameter. For crutching soaps that are somewhat thinner, such as are usually made in England, the





FIGS. 73, 74.—CRUTCHING VESSEL.

crutching machinery designed by Neill & Sons, St. Helens, Lancashire, is suitable.

One of the earliest methods of cheapening, hardening, and increasing the detergent powers of soap was that proposed by Dr. Normandy, who mixed "neat" soap with crystallised sodium sulphate, previously melted in its own water of crystallisation. The salt re-crystallised in the soap as it cooled, and the soap was thereby considerably hardened, so that it wore better in the wash-tub when rubbed upon clothes, and in this way its detergent power was mechanically increased, although sodium sulphate as such, being a neutral salt, had no detergent power of its own, and its addition to soap really diminished chemically the percentage of soda available for washing. These soaps usually effloresced with a white powder, and gradually fell out of use, especially as raw fatty matters became cheaper.

It was then discovered that the addition of sodium carbonate, or "sal soda," has a remarkable effect in stiffening and hardening soaps to which it is added in a state of strong solution; it also increases chemically their detergent power. This process is very largely employed in America; the amount of soda used depends upon the raw materials from which the soap is made, and upon the quality of the desired product; a very usual proportion is 1 cwt. sodium carbonate crystals (melted) to every ton of soap. Not unfrequently a solution of pearl-ash (impure potassium carbonate) and common salt, mixed in varying proportions, at a sp. gr. of about 52°–64° Tw. (30°–35° B.), is used for a similar purpose.

Silicated Soaps.—The discovery of methods of manufacturing on a large scale soluble potassium and sodium silicates, gave a very important impetus to the soap trade, since these substances are peculiarly suitable for the purposes now being described. Their first application on a large scale was the production of soaps by Christr. Thomas & Bros., of Broad Plain Soap and Candle Works, Bristol, containing "both silicate and sulphate of soda," and by these means they

were able to produce, and patented in 1856, a soap of very great detergent power, which could be sold retail at less than the duty on soap, which had been removed a few years previously. It is usually supposed, however, that the value of silicated soaps was first publicly and officially recognised at the International Exhibition of 1862, when a prize medal was awarded to W. Gossage and Sons, Widnes Soapery, for their samples.

Sodium silicate may be mixed with almost any kind of soap, but the strength of the solution employed must be varied according to circumstances. Very weak solutions are often added to "neat yellow soaps," and when employed in this way it is a good general rule, *ceteris paribus*, to increase the sp. gr. of the solution with the percentage of it employed. Thus, if it be desired to increase the quantity of water in a "neat" soap by 4–5 per cent., a solution at 7½° Tw. (5° B.), when cold will be suitable; while if the quantity of water is to be increased to a total of 50 per cent., a stronger solution, say 15°–18° Tw. (10°–12° B.) is required. This kind, technically known as "run soap," was at one time largely made in America, and still is in England, under the name of "London pale" in the south and west, and of "Primrose" in the north. Such soaps are of the consistency of thin treacle when mixed, at say 160°–170° F. (71°–77° C.), and are apt to disappear rapidly in hot water, as well as to lose weight when kept. They are usually subjected to the storing or case-hardening process described on p. 188.

A more legitimate application of sodium silicate is to mix varying quantities of the concentrated solution with "neat" yellow or curd soaps. This treatment makes yellow soaps much stiffer, and in many cases, by hardening them, adds to their durability. About 5 per cent. of the solution at 140° Tw. (59½° B.), is a suitable quantity, and has much the same effect as the addition of the 5 per cent. of sodium carbonate crystals before described. Much larger quantities than 5 per cent. may be used, but soap so treated is apt to disintegrate unpleasantly in use, as well as to feel very

harsh to the hands of the consumer. Curd soaps are sold in England with which 15 or even 20 per cent. of sodium silicate at 140° Tw. (59½° B.) have been mixed. These large quantities considerably increase the "soda available for washing," as given by the alkalimetric test (see Soap Analysis, Chap. XI.).

In connection with silicated soaps, the following quotation from C. F. Cross's lecture on Soap at the International Health Exhibition of 1884, deserves attention:—

"The solution of sodium silicate is inferior in lathering properties; its detergent action is not aided, as in the case of the soaps, by a softening action of the acid constituent upon the surface to be cleaned; and lastly, it cannot be brought into a form so universally convenient as that of soap, existing only as a glassy solid or a viscous liquid. Therefore it can only be used in a state of solution, or when incorporated with a sufficient proportion of soap to allow the physical properties of the latter to preponderate. These considerations will enable us to appreciate the right side of the 'silicated soap' question. The wrong side consists, not in production by means of the silicate of a detergent of lower quality, but in not selling it at a proportionately low price. A silicated soap is an advantage to commerce, fo there is much work which it can perform as well as, if not better than, soaps of higher quality. But let there be fair division of the advantage between manufacturer and consumer, and the millenium will not suffer postponement."

Blue, Grey, and Red Mottled Soaps.—These come under the head of silicated soaps, and are thus made. Two coppers are required, an ordinary steam-copper for the first stage, and a fire-copper for the later stages. In the steam-copper, the raw materials are saponified, and fitted rather open. The fat-mixtures employed are usually vegetable oils, and almost always (though not necessarily) contain a fair proportion of cocoanut or palm-kernel oil. When first introduced from Germany, these soaps were made from well-bleached palm oil and cocoanut oil, in such proportions as 3 palm to 2 coco-

nut, or 2 to 1, or even 3 to 1. Latterly, however, palm-kernel has supplanted cocoanut oil, and some of the palm has been replaced by refined cottonseed oil. The choice of materials is very much guided by their cost. The fitted soap is shifted off its nigre into the fire-copper, and to every 1000 lb. of it is added about 250 lb. solution of sodium silicate at about 32½° Tw. (20° B.); the exact sp. gr. depends chiefly upon the proportion of palm-kernel to other oils. The whole is then boiled together with steam and fire, to thoroughly incorporate the mass; when this is complete, the steam is stopped off, and the appearance of the copper is examined. Practice and experience, assisted by chemical analysis, can alone decide when the soap is in "mottling condition"; in that state, it should have about 45 per cent. (or less) of fatty acids, and 0·5–1·0 per cent. of sodium chloride, according to the raw materials employed. A good physical test is to take a layer out rapidly upon a cold trowel, and observe its appearance, and the time required for it to "set." A shiny appearance on its surface indicates a deficiency, a frosty appearance, an excess, of mineral salts; if it sets too quickly, and is shiny, more sodium chloride must be added; if it appear frosty, and is long in setting, enough mineral salts are present. So delicate is the process, that the addition of 1 lb. salt per ton of soap at this stage will entirely alter the appearance of the mottling when cold. When it is in mottling condition, the mineral substance used as mottling is mixed with a small quantity of water, and sprinkled into the copper; it is there thoroughly incorporated with the soap by a few minutes' boiling, and the soap is then transferred as rapidly as possible to *wooden* frames, which are carefully covered up when full, and kept as warm as possible, to allow time for the "mottle to strike." For blue mottle, 5–10 lb. artificial ultramarine per ton of soap are used, for grey, 1–3 lb. finely levigated manganese oxide. If the soap be cooled rapidly, it will be of a homogeneous blue or grey colour; slow undisturbed cooling is essential to these soaps, and once in the frame, they should never be touched

until they are quite cold; it was for them that Gossage devised the pneumatic method of emptying coppers (p. 183), but a centrifugal pump answers as well. It requires greater skill to make good "blue-mottled" soap than for any other kind, and the manufacture is in the hands of a few large firms.

Another mode of producing these soaps is to make a portion of the fat employed (usually all the coconut oil, with or without some portion of the other oils) into hydrated soap (p. 169); the remainder of the fatty matter is made either into a "fitted soap" or a "flat curd" soap, and then transferred to the hydrate previously prepared in another copper; after both are incorporated by thorough boiling, the soap is finished as before directed. This method, for which Blake & Maxwell and C. N. Kottula had various patents, was introduced into England from Germany by the last named about 35 years ago; it is said to produce a more solid and close soap from the same materials than any other method, but when a blue-mottled has to be made, the greatest care must be used to allow no impurities in the materials for the hydrated soap, or the brilliancy of the mottling will be seriously interfered with.

It may be observed here that in these soaps, the mottling, so difficult to produce, is a matter of appearance merely, and that soap with a plain white ground would wash just as well. The remarks of Cross, upon mottled soaps, in his Health Exhibition lecture quoted on p. 200, are very much to the point in this case. Speaking of naturally mottled curd soaps, he says:—

"Still more meretricious methods have been followed for imitating this *natural* marbling of soaps, not so much for the sake of the particular effect, but because mottled soaps are necessarily of high quality, and the next thing to possessing this quality is to possess the appearance which is its more obvious criterion. . . . The practice of artificially mottling inferior soaps—even though there be not the slightest intention to defraud—is not to be com-

mended. It is difficult to see any useful purpose which it subserves, and it certainly cannot be defended on art grounds. We would therefore ask consumers to aid in breaking down a bad custom—bad, because it wears an *appearance* of evil intent—by avoiding artificially mottled soaps. We would more especially instance the blue mottled soaps in which the marbling is produced by means of ultramarine blue incorporated into the soap-paste. It requires very little insight to enable the buyer to detect an artificially marbled soap, and we hope no further showing to induce him to reject it when detected."

To this emphatic condemnation may be appended the published remarks of another soap-manufacturer, Leopold Field, who, in speaking of the various classes of soaps and of Castile soap and the common red, blue, and grey mottled soaps in particular, says:—

"The two last are really only varieties of the others, and well exemplify the manner in which manufacturers have to fall in with the public prejudice to the public detriment.

"From time immemorial the soaps made in the south of France and the north of Spain (Marseilles and Castile) have enjoyed the highest reputation, and deservedly so; inasmuch as, at least till lately, they were prepared from vegetable oils and natural alkalies containing enough potash to give a plasticity and smoothness which was lacking in pure soda soaps. Now, as these natural alkalies contained a certain amount of mineral matter (oxides and sulphides of iron and manganese), the soaps, during making, developed coloured streaks and blotches, thus acquiring a marbled appearance. In a thin watery soap, this colouring matter would have sunk to the bottom, and hence the mottled appearance was held to be a proof of excellence. With fine logic, therefore, the public came to the conclusion that because Castile soap, which was good, was mottled, therefore all mottled soap must be good. Consequently the manufacture of 'mottled soap' has become an art of itself, the object naturally being to get as much mineral matter in as poor a

soap as may be, and in this, as in most branches of adulterative art, the German shines supreme." [The manufacture of these "artificial mottled" soaps was introduced into England by a German, C. N. Kottula.]

Sundry Fillings.—Sometimes the silicate of soda is replaced by aluminate of soda in scouring soaps.

Borax is commonly added to laundry soaps, as it possesses considerable detergent power, is moderately disinfectant, exerts no injurious action on textile fabrics, and operates to some extent as a bleaching agent.

Petroleum or paraffin is a useful addition to laundry soaps when dealing with greasy linen, as it notably facilitates the removal of greasy matters, and thus reduces labour and preserves the articles from excessive rubbing or scrubbing. Barbieux and Rosier proceed by melting about 15 per cent. of fatty matter in a water bath and mixing it with the mineral oil by stirring; 2 parts of this and 3 of fatty matter are combined ready for saponification in the usual way. Green incorporates mineral oils thus: to 1 cwt. mineral oil is added 5–10 per cent. of a saturated alcoholic solution of potassium subchloride and caustic soda, with agitation and boiling; then 1 cwt. sodium silicate and 3 cwt. water, and renew the boiling; then 2–3 cwt. of soap in flakes or shreds, by degrees, and with continued stirring. When melting and admixture are complete, the mass is run into frames.

Lorberg adds 10 per cent. of a solution of gluten and caustic alkali, made thus: to a solution of caustic alkali at 32½° Tw. (20° B.) is added as much bran or gluten as it will take up; after some hours' digestion, the mass is filtered, and the filtrate is employed.

Some of the worst examples of filling are recorded in patent literature. Thus, Lavandier secured letters patent for incorporating 50–100 per cent. of kaolin. Waller obtained similar rights for adding sawdust. Armstrong and Bostock incorporate 33 per cent. of glucose. Higgins introduces an unusually large percentage of rosin with hard soaps. Thus for a cheap laundry soap 10,000 lb. of fat is saponified in the

usual way with caustic soda at 52½° Tw. (30° B.); after the "grease change," 10,000 lb. rosin is added and likewise saponified, a total of 6000 lb. of caustic soda being needed. Saponification being complete, and the mixture still hot and fluid, before framing, 2 per cent. stearic acid or 3 per cent. stearine, hot and fluid, is well crutched into the body, which is then framed as usual.

CHAPTER X.

SPECIAL SOAPS.

A FEW of these, including the commoner kind of scented soaps, will now be considered. Cheap toilet soaps are thus made on a large scale. For "Honey soap," a good "neat" yellow soap is taken, and an aqueous solution of some yellow dye, such as may be obtained from the dealers in aniline colours, is mixed with it; 5 per cent. sodium carbonate crystals, or 5 per cent. sodium silicate at 140° Tw. (59½° B.), is then added to stiffen it; the whole is crutched, and scented by the addition at as low a temperature as possible of as much citronella-oil as is deemed necessary. For "Brown Almond soap," an inferior grade of yellow soap is similarly treated, and scented with about 10 lb. of mirbane (i. e. nitrobenzol) or artificial almond-oil to the ton of soap. When cold, these soaps are cut into bars or cakes, superficially dried, and stamped with one of the foot-power or steam stamping-machines (p. 192). A similar method may be employed for the incorporation of any cheap scent or colour, or other desired admixture. The apparatus employed necessarily depends upon the quantity operated on at one time, and for the choice of this, the hints given on p. 196 are sufficient.

Floating Soaps.—So-called "floating" soaps, as first introduced, were made by dissolving soaps in a small quantity of water, and agitating the solution violently in contact with air. They are now prepared according to various methods, of which two will suffice for detailed description—(*a*) preparation from fresh materials, and (*b*) preparation from

trimmings of cocoanut oil soap. This latter will probably give a very welcome opportunity to many manufacturers to advantageously dispose of the heaps of trimmings often left over.

The following is a formula for preparing a white floating soap from fresh materials. The colour of the soap will of course depend largely on the quality of the oil used :—

Cocoanut oil	88 lb.
Soda lye, 74° Tw. (38° B.)	46·2 „
Potash lye, 42¼° Tw. (25° B.)	2·2 „

Melt the cocoanut oil in the usual manner, filter into a capacious jacketed kettle, or one placed in a water-bath, and heat to about 122° F. (50° C.). Then add the lye, stir well for about 10 minutes, and cover up the kettle. Allow to saponify, and thoroughly stir again. The soap will now have the appearance of fine woolly grains.

In the foregoing process but little fire or steam is necessary; 22 lb. of well-warmed calcium chloride solution of 32½° Tw. (20° B.) and 88 lb. of hot water are now gradually added with constant stirring to the curd in the kettle. The soap is worked up thoroughly to complete solution, but very little heat is required, as it is not necessary to make the soap boil.

After obtaining complete solution, take a lye cylinder full of the soap solution from the kettle, allow it to cool to 77° F. (25° C.), and sink a lye hydrometer in the liquid, when this will indicate a density of 106° Tw. (50° B.). This particular degree will yield a floating soap having a medium weight.

The soap solution is then allowed to cool to 77° F. (25° C.), and a stirring kettle is filled about one-third full with the cooled soap. This aqueous fluid mass is stirred vigorously until transformed to a stiff foam, and is then put into the frames at once.

The prescribed temperature of 77° F. (25° C.) must be carefully adhered to, for if heated to a higher temperature, say 100° F. (38° C.), or over, much more time will be required to work up the liquid into a permanent form, and through

the long stirring the foam would be so puffed out that the resulting soap will be too light. On the contrary, if allowed to cool too much, the soap obtained will be too heavy, because the formation of the foam takes place too rapidly, and the soap is not allowed sufficient time to swell in the kettle.

Floating soap should not be dried in a warm room, nor in a drying oven, as, if this is done, the soap will shrink a great deal and become fissured. It is better to allow the entire block, as it comes out of the form, to stand for several weeks in an airy, light place; then cut into tables, allow them to dry for several days, and finally cut up into bars or cakes.

Another process, that of making floating soap from trimmings, is quite simple. For instance, place 220 lb. of the trimmings or scraps from coconut oil soap in a jacketed kettle or in a water-bath. To dissolve this, about 33 lb. of potassium chloride solution of 32½° Tw. (20° B.) and about 132–154 lb. of water should be added to the scraps in the kettle, the quantity of solution and water required being of course dependent on the degree to which the scraps have dried out.

Considerable heat is applied at first, and the scraps are diligently broken up to facilitate their solution. Strips and cubes of soap should have been previously passed through a planing machine. When very old, dry scraps are used, it will frequently prove very difficult to effect their solution. In this case, solution can be accelerated by strewing over the above quantity of soap 2–4½ lb. of salt.

The trimmings of coconut oil soap mentioned in the above process should not be from "filled" soap, as such, filled for instance with water glass and soda crystals, are not suitable for floating soap. The material used for filling renders the soap brittle and coarse, and when cut and planed the surfaces of the bars and cakes do not become smooth. When used in too large quantities, salt causes the same result in floating soaps. These "filling" solutions have also an influence when measuring the degree of density of the soap solution.

If it is desirable to work in scraps of floating soap, they

should be added to the already dissolved soap mass in the kettle, together with the necessary amount of water. They will then dissolve very easily.

When putting the soap solution into the kettle for stirring, it is advisable to allow it to pass through a fine sieve to get rid of any bits of wood, paper, or dirt which may be present.

As to the colour of floating soap, it may be said that white is by far the favourite colour.

By using small quantities of tallow or lard in making floating soaps, they are rendered firmer and more solid, as well as more economical in use.

The perfume should be added to the soap in the stirring kettle before the soap begins to form a thick foam. The chief perfumes used are lavender, caraway, white thyme, and fennel oils.

This soap can be cut with a fine steel wire; for planing the bars, a very fine sharp plane is required.

Disinfectant Soaps.—In few ways can disinfectants be so agreeably applied to the skin as when incorporated with soap. One of the last introduced, though probably one of the most efficacious, is thymol soap—first made by Ferris & Co., Bristol. Thymol is a non-poisonous (herein differing from carbolic acid) crystal, about 8 times as powerful an antiseptic and disinfectant as carbolic acid, and is probably the only substance that combines disinfecting properties with a really pleasant smell, that of thyme. The best mode of incorporating thymol and phenol (i. e. carbolic acid) with soap is a trade secret; Morfit, however, states that carbolic soaps are best made by his process (p. 153), using as a basis hot-pressed fat-acid cake, on account of the tendency of carbolic acid to soften the soap-paste.

Carbolic soaps are made in great variety and in large quantities by F. C. Calvert & Co., of Manchester, whose products contain specified definite quantities of carbolic acid of various qualities. Their "medical" soap contains 20 per cent. pure crystallised carbolic acid; their toilet and household soaps, 10 per cent.; their domestic soap, 8 per cent.;

and their "No. 5" or "scouring" soap, 4 per cent. liquid carbolic and cresylic acids. The comparative antiseptic power of soaps may be tested by adding equal weights, in solution, to equal weights of flour-paste, and, after exposing these to the air under identical conditions, noting the day on which mould first appears on each. The so-called "coal-tar" soap or "sapo carbonis detergens," owes its disinfecting properties to a small quantity of carbolic acid in the coal-tar.

It may be assumed as a pretty safe working rule, that almost any desired substance, whether soluble in water or not, can be mixed in this manner with soap, provided that (1) it is not too volatile at the lowest temperature at which the soap can be manipulated; (2) that if insoluble in water or alcohol it is in a state of very fine subdivision; (3) that it contains neither enough acid to decompose any portion of the soap, nor any mineral bases which would cause double decomposition with the sodium salts of fatty acids.

Thus Clever incorporates terebene, copper alum or iron alum; Robottom adds borax or other borates; and others select camphoraceous products obtained by artificial oxidation of turpentine oil, such as "sanitas."

Hard-water Soaps.—In order to reduce the waste of soaps in contact with hard waters, through double decomposition brought about by the lime and magnesia salts, Funk and Eltze, in 1877, patented the use of sodium phosphate; and in 1890, Grimshaw claimed novelty in the addition of "an alkaline phosphate," with the object of forming calcium and magnesium phosphates, instead of lime and magnesia soaps, which are insoluble in water and therefore wasted.

Sand Soap.—Under this heading, occur a number of soaps in which it is sought to unite the chemical action of soap with the mechanical aid afforded by sand in scouring. According to C. Roth (*Seifensieder Zeitung*, Nov. 21, 1884), as much as 70 per cent. of clean sand or powdered quartz is sometimes mixed with soap-paste, and experiments showed that such soap had no disagreeable effect on the hands when

used as a detergent. In a similar way, soap is made the vehicle of many substances to be applied to the skin, medicinally or otherwise, or in any cleansing process. All these should be incorporated with "neat" soaps, freshly made or remelted, at as low a temperature as possible. Some form of soap is not unfrequently the basis of polishing pastes.

It is obvious that many abrasive substances besides sand can be incorporated in soap, such as fuller's earth, pumice, brickdust, emery, &c., in all proportions up to the limit of cohesion of the mass. Even some toilet soaps are so prepared for application to the teeth, substituting precipitated chalk, cuttlefish bone, &c., for the commoner forms of abrasive material.

Cold-water Soaps.—This term, which has made its appearance within the last few years, was at first confined to soaps made from very soft fatty materials, but containing a very small amount of water; such, for instance, as those produced by Morfit's process. Similar products are also very largely made by artificially depriving "neat soap," fitted in the ordinary way, of about $\frac{1}{3}$ of the 30–32 per cent. of water which it naturally contains. This is done by exposing it to prolonged fire-heat in a vessel in which it can be thoroughly agitated at the same time; occasionally it is also stiffened by the addition of sodium carbonate or silicate, as described above. These soaps are sold at a low rate, and, from their great dryness, may be kept indefinitely without losing weight, a property possessed by scarcely any other household soap; being perfectly pure soap, they are truly economical, provided they are not used with hot water. Latterly, however, the use of the term has been appropriated by makers of heavily-watered soaps, which run away in hot water. Hence, in this case also, the consumer should be careful what kind of soap he buys under this name.

Soap Powders.—These always contain, besides powdered dried soap, a large percentage of sodium carbonate, generally in the form of dried soda crystals. They may be prepared in several ways, thus:—

(*a*) Anhydrous sodium carbonate or soda ash is added to a "clear-boiled" soap-paste, and after thorough mixing, the somewhat stiff material is drawn off into cooling frames. The cold and hard soap thus obtained is then finely ground.

(*b*) Soda crystals and soap are melted together and then treated in the above manner; but this plan is advantageous only when soap scraps can be had.

A suitable apparatus for conducting the operation consists of a wrought-iron vessel with a strong agitator contained in an interior cast-iron vessel, which can be cooled by water circulated in the outer vessel. The liquid soap is cooled while the soda ash is slowly added and completely dissolved. During the grinding process, care must be taken not to overheat and thus soften the product.

The composition of soap powders varies considerably. Only a small proportion of rosin soap can be used, as such soap is sticky, and cannot be powdered. Olein soap may be used with advantage, and the olein may be saponified with sodium carbonate instead of the more expensive caustic lyes.

As a small quantity of free chlorine is not objectionable in soap-powders, dark coloured materials such as bone fat, fish oils, &c., may be used for making the soap, with the addition of a small quantity of bleaching powder.

To some soap powders 2 to 5 per cent. of sodium silicate is added.

A good washing powder should contain 30–35 per cent. fatty acids, 30–35 per cent. sodium carbonate, 30–40 per cent. water.

The inferior powders, containing only 5–10 per cent. fatty acids, should not be used for the laundry; they are only serviceable for scrubbing purposes.

There is a soap powder in the market prepared by treating linseed with caustic soda directly. This soap contains certain impurities derived from the seeds, which lather freely, and thus, when the powder is used, gives the impression of more genuine soap being contained in the powder than is actually the case.

For washing printers' blocks and types, a powder is recommended containing only a small quantity of combined fatty acids, but 10–15 per cent. caustic soda.

The so-called "bleaching soda" consists of 80 parts soda crystals and 20 parts soda silicate.

The composition of some American washing powders was found to be—

	a	*b*	*c*	*d*
Sodium carbonate	45·2	26·9	49·2	46·6
Fatty acids	26·4	44·0	25·6	25·7
Combined soda	3·1	3·4	3·5	2·6
Water	23·7	8·8	19·1	24·9
Fine sand	..	16·3	..	..

They are therefore mixtures of dried soap and washing soda, and may be used in conjunction with soap to soften hard water, but if used alone must be injurious to textile fabrics. Borax might be added instead of the soda, apparently with advantage.

Manufacturers' Soaps.—The various kinds of household soaps having now been described, a few remarks will be made upon the soda-soaps suitable for various manufacturing purposes. Most of these are dissolved in water for use, and hence it is immaterial into what sized bar they are cut. Care, however, should be taken that they *are* dissolved; a case occurred in the writer's knowledge when the quality of a soap was much complained of, as producing greenish stains upon black cloth. The soap-maker asserted his ignorance of anything deleterious in the soap, and subsequent investigations showed that the cloth-manufacturer's workman, instead of completely dissolving the soap, had impregnated the cloth with a solution containing *undissolved pieces*, and the soda in these, not unnaturally, affected locally the indigo and logwood with which the cloth had been dyed.

For ordinary scouring purposes, there are few better soaps

than the old-fashioned curd-mottled; many others, however, are used, such as curd soaps boiled very dry, made from cheap and inferior greases, cotton-oil foots, &c., and fitted soaps from greases and black rosin. For scouring goods of finer quality, a white curd soap from tallow, or tallow and lard, with, or without, the addition of a small quantity of coconut oil, is used, or a curd soap from olive or cottonseed oils, or a mixture of both. The soaps made on Morfit's plan (p. 153), are also good scouring soaps. As a rule, traces of unsaponified fat (or indeed any extraneous material) are very deleterious in manufacturers' soaps, which, under ordinary circumstances, should contain a very slight excess (as curd and mottled soaps always do) of caustic soda. When for any purpose, an absolutely neutral soap is required, as e. g. where delicate dyes are employed, either a "finely-fitted" soap should be used, or a curd soap from which the caustic lye has been pumped off, and the soap finished by boiling on brine.

A very ingenious mode of making soaps of any kind absolutely neutral was introduced in 1885 by Dr. C. R. Alder Wright. Some salt of ammonia, such as ammonium sulphate or chloride, is mixed up with the soap paste while hot. The caustic soda present turns out the ammonia (producing sodium sulphate or chloride), and much of the ammonia is volatilised at once by the heat of the mass, while the remainder escapes after the soap has been cut up and exposed to the air for some days.

According to Crace-Calvert, soaps for dyers' use are not indiscriminately applicable to all colours. To produce the maximum effect in brightening the shade, the soap should have the following composition:—

	For Madder Purples.	For Madder Pinks.
Fatty acids	64·4	59·23
Soda	5·6	6·77
Water	34·0	34·00

The problems to be dealt with in the use of soap by dyers, however, belong more properly to the technology of that

industry, and probably cannot be settled by so elementary an analysis as this.* Such questions may be involved as the proportion of soda to fat acids of a particular molecular weight (*vide* Chap. XI.)

For some purposes, a soap whose solution will remain liquid at a low temperature is required; such soaps are well made by Morfit's process, and should contain large quantities of oleic acid. For "fulling," this soap is often employed, mixed with curd soap made from unbleached palm oil only.

Much has been written about the frauds practised by unprincipled soap-makers upon manufacturers using soap, and the latter have been advised, in self-defence, to make their own soaps. Reasons have been given (p. 168) against this course; it is much to be desired, however, that soap-*users* would take soap-*makers* personally more into their confidence in explaining their requirements, and would themselves superintend (and not leave to their foremen) any experiments made on the working of different kinds of soap. A system, too, on which manufacturers' soaps should be sold, guaranteed to contain a given percentage of fatty matter of a definite quality, with its full equivalent of soda, is greatly needed. Such arrangements, if carried out, would very probably put this trade upon a far better footing than it is at present.

There is scarcely a single textile fabric of any material manufactured which does not pass at one stage or another through an operation in which soap plays a part. Seeing this, it is evident that the production of textile soaps must be an important industry; in fact, there are firms who manufacture soaps for this purpose alone. The objects for which soap is employed are as varied as the textile fabrics themselves. The quality of the soap depends very largely on the kind of fat employed, which may be of animal or vegetable origin, and also upon the care taken in the process of making;

* The question has recently received much attention in the Dyeing School attached to the Yorkshire College, Leeds, where the pupils receive special instruction in this subject.

good materials may be spoiled by faulty manipulation in soap-boiling as in other work. The following observations are from a specially contributed article on the subject in the 'Chemical Trade Journal,' of January 19, 1895.

Wool Soaps.—Soap is employed in three stages, if not more, in the manufacture of the raw wool fibre into cloth. The very first operation that wool undergoes is that of scouring to free it from grease and dirt; this is done either by treating in a warm soap liquor, or in a warm bath of carbonate of soda or potash. Some wool-scourers use a mixture of both. Many wool-scourers use a very common kind of soap for this purpose, made with soda from poor qualities of fat, and consequently dark coloured, as well as often being imperfectly saponified. Bone tallow, tripe tallow, low grade palm oil, and oleic acid are much used in making such soaps. They are best left slightly alkaline, the excess of alkali being neutralised by the grease of the wool. Too great an excess of alkali, however, should be avoided, as it tends to make the wool harsh to the touch, and if soda has been used, yellow in colour. Potash gives the best soap for wool, inasmuch as the alkali has greater scouring properties, while it has little action on the wool fibre, the latter being left softer and more lustrous in appearance. A good sample of a soap for scouring wool will contain 62 per cent. of fat acids, 8 per cent. of combined alkali, ½ per cent. of free alkali, ¼ per cent. of free fat, 29¼ per cent. of water. A soft soap will contain 50 per cent. of water, ½ per cent. of free alkali, ½ per cent. of free fat, 6 per cent. combined alkali, 43 per cent. of fat.

The soap should be fairly soluble, so that a good strong soap liquor can be made without being thick. Tallow and palm oil make a hard soap not readily soluble in water.

After the wool has been scoured to free it from grease and dirt, it is necessary to oil the wool before it can be spun and woven. Before the yarn or cloth thus made can be dyed and finished, it is needful that this oil be extracted, and for this purpose nothing has been found better than soap. Generally the same kind of soap is used as for the scouring. A very

favourite soap, because it is cheap, is one made from bone tallow and cotton oil; this soap lathers pretty freely, and it is usually considered that a soap which makes a good lather clears the wool from the oil fairly well. Another good soap is made from palm and cotton oil. In the case of soaps for removing oil from wool, it is of very great importance to use one of good quality, or it may not remove all the oil, thus causing uneven shades in the subsequent dyeing operations. It is also important that the soap itself should contain nothing which would be liable to fix upon the wool and interfere with the dyeing. The fats employed ought to be quite pure, and free from any unsaponifiable matter. For this reason bone tallow is not good, as it contains some phosphate of lime and carbonate of lime, as well as animal matter. The lime has a tendency to form lime soaps, which eventually get on to the wool, forming grease spots. Recovered greases should not be used, as they often contain perceptible quantities of unsaponifiable matters. A soap made from pure fat has none of these defects.

Woollen cloths after being woven and dyed have to be milled or fulled. This operation causes the fibres to felt together, and so make a more durable and less pervious cloth. This milling is also accomplished by working the goods in a soap liquor.

The soap here has very little actual work to do, its principal function being that of a lubricant facilitating the movement of the fibres one over the other and assisting the process of felting. But even if the soap has not much actual work to do, there are still some things it must not do. It must not cause the colours of any dyed material to "bleed," nor must it change the tone, or dull the hue of any dyed goods. This can only be avoided by the employment of a soap as free from alkali as possible. A perfectly neutral soap is best. Such a soap requires care in making. The soap too ought to have good lathering properties, and be pretty freely soluble in water. It is therefore best made from olive, cotton, or other similar oils. Tallow and palm

oil do not yield good lathering soaps. A potash soft soap is the best, but it is rather more difficult to make of the desired degree of neutrality.

Cop Soap is used in the spinning of cotton. It has been found that, by smearing the bottom of the cop spindles with a little soap, the bottom of the cop is formed better, due to the threads adhering together. For this purpose, a soft soap is found to be the best. It ought to be free from colour, hence these soaps should be made from tallow, olive, ground nut, or coconut oils. They should also be free from mineral oil or paraffin wax, as such have a tendency to soak into the cotton fibre, and cannot readily be extracted, thus causing stains when being bleached or dyed.

Calico Printers' Soap.—The calico printer makes great use of soap. He employs it first to cleanse the calico from the gum or other thickening agents which he is obliged to add to his printing colours. Then it often has a brightening effect, as with alizarine colours.

Most calico printers prefer to employ a soap made from palm oil for this purpose, chiefly on account of the fact that palm oil makes a soap which has a pleasant and agreeable odour, and does not easily turn rancid. Tallow, coconut oil, and castor oil, while making soaps which are satisfactory in other respects, often turn rancid, especially in the case of the last two oils. The soap must be well made, and, above all, must not contain more than traces of free fat and free alkali, the latter acting upon the colours and changing their tints. A good printers' soap will have a composition approximating to the following:—Water, 23·5 per cent.; alkali as soap, 9·0 per cent.; alkali, free, 0·2 per cent.; fat, free, 0·2 per cent.; fat as soap, 67·1 per cent.

Olive oil soft soaps are also used by calico printers for much the same purpose; these are easily made by boiling olive oil and potash together.

Softenings.—In the finishing of cotton cloths, what are known as "softenings" are much used. These are nothing more than highly watered soaps. The writer has found as

much as 80 per cent. of water in such materials. This quantity is rather excessive, but from 50 to 60 per cent. is permissible. Softenings vary very much in composition: some are simply made by taking an ordinary tallow or bleached palm oil soap and boiling it in water until melted, then allowing to cool. Others are made direct from tallow, palm oil, or coconut oil, and then thinned down with water to the required degree of consistency. Another form is made from coconut oil by means of a cold process. Sometimes paraffin wax is added. Softenings should be white in colour, they therefore ought to be made from tallow, bleached palm oil, coconut oil, castor oil, or other white oils. They ought to be neutral in properties, so that they do not affect the colour of any printed or dyed goods they may be brought in contact with.

Silk Soaps.—For degumming silk nothing has been found better than a good olive oil soap. A silk soap should possess the following properties. It ought to be neutral, inasmuch as alkali tends to make the silk somewhat harsh, and to reduce its lustre; and it should be easily soluble. The soaps used in the boiling off of silk must be well-made curd soaps, using as slight an excess of alkali as possible, and well salted out. The soap must also be capable of being readily washed out of the silk. A soap well made with potash and olive oil will be found to give excellent results in the boiling off of silk.

Way adds alumina phosphate and caustic soda to soaps for dressing woollen and silken fabrics.

Scharr's liquid soap for scouring yarns, &c., is made as follows:—For 100 gal. of soap, mix 66 gal. water, 12–21 lb. linseed, 2 lb. farina starch, 13 lb. calcined potash, 162 lb. refined soda ash, 12 lb. rosin, 4 lb. borax, 3 lb. sal ammoniac, 2 gal. olein, 3 qt. spirit of turpentine, and 2 qt. liquid ammonia at ·880°, with 4–6 qt. water. His soap cream for dressing textiles is compounded thus. To make 100 gal. requires 55 lb. Marseilles soap, 6¼ lb. sodium carbonate, 6¼ lb. pearlash, 3½ lb. borax, 175 lb. olive oil, 2½ lb. Japanese isinglass, 1½ lb.

tragacanth, ½ lb. salicylic acid, and 37 lb. glycerin. (*a*) The soap, soda, pearlash, and borax are boiled 2 hours in 40 gal. of water; the olive oil is added, and boiling is continued; (*b*) meantime the isinglass and tragacanth are steeped and boiled, the salicylic acid and glycerin being introduced when solution is complete. When *b* is cool, *a* and *b* are well mixed together.

FINE TOILET SOAPS.

Three distinct processes are in vogue for the fabrication of these, according to the quality of the product desired. For the commoner kinds, the basis is a good grade of fitted yellow soap, taken direct from the copper, or re-melted in a small steam-jacketed pan, or in a Whitaker re-melter, provided with continuous coils of steam-pipe. To this are added (1) suitable colouring matter, in a soluble form if possible, such as some aniline dye; (2) some mineral salts, as sodium or potassium carbonate, salts of tartar, &c. (silicates should be avoided), to stiffen and "close" the soap, usually about 5 per cent. in strong solution; (3) at as low a temperature as possible, the perfume. When cold, the soap is cut up into slabs, bars, and cakes, dried, and stamped, as previously described.

Toilet soaps have been treated at length by Dr. C. R. Alder Wright in a Cantor Lecture, published by the Society of Arts, in 1884, from which much information may be gleaned. Properly speaking, a "toilet" soap differs from the ordinary household soap only in the quality of the materials used in the manufacture, and the care and skill employed in so conducting the operation and proportioning the ingredients to one another, or in subsequently purifying and refining the crude product, as to obtain, as the final result, *a material in which the alkali originally used has been all but perfectly transformed into true soap*, leaving none uncombined with the fatty matters employed. But with a large portion of the toilet soaps sent into the market, such processes are not adopted; on the contrary, methods of treating the soap are

largely used *which add to the alkalinity*, thereby greatly impairing its value from the point of view of suitability for habitual use by persons possessing tender and sensitive skins. The reason for the adoption of these methods is either for the sake of improving the grain or texture of the mass (an improvement dearly purchased at the expense of deterioration in quality), or of communicating greater hardness and durability. The first result is usually sought to be effected by "pearlashing," i. e. adding to the melted mass a proportion of pearlash (or refined potashes), which brings about a remarkable chemical change, viz. the transformation of soda soap into potash soap, with simultaneous production of carbonate of soda instead of carbonate of potash; and the admixture of these products with the original mass causes a marked alteration in its physical character. The second result is often attained by similarly incorporating into the mass "soda crystals," which addition causes (especially with certain kinds of fatty acid soaps) a notable stiffening and hardening of the whole, so that a firm mass results, even if a moderately large amount of water be present. The same effect can also be produced by the employment of certain neutral salts, e. g. sulphate of soda and common salt. The practices of "watering" and "closing up" by addition of saline matters are now carried out to such an extent, and so ingeniously as to be almost a fine art, so that there is a proverbial expression concerning this class of trade, referring to "making water stand upright" as a main source of profit; and as regards the amount of foreign matters that can thus be incorporated (especially when cocoanut oil is freely used as part of the fatty matter, this substance lending itself to the practice better than almost any other), it is by no means uncommon to find on analysis less than one-sixth (16·7 per cent.) to be actual soap, the balance being water with saline matter added, to close or harden the soap. Soaps that have been heavily watered are always apt to get out of shape by exposure to air, through shrinkage in drying; and often to become simultaneously covered with a white saline efflorescence,

caused firstly by the "sweating" out of liquid from the interior, and, secondly, by the evaporation of the water, leaving behind a more or less crystallised mass of the salts used for hardening, which were dissolved in the liquid. When any noticeable efflorescence is produced on a tablet on keeping, there is at least a high degree of probability that an undue amount of alkalinity is possessed by the soap.

A few formulæ for perfumes are here given, calculated in each case for 100 lb. soap:—

Almond Soap.

	oz.
Oil of bitter almonds	12
„ lemon	4

Brown Windsor Soap.

Oil of cinnamon	4
„ cloves	1
„ caraway	1
„ sassafras	2
„ bergamot	2

Or—

Oil of bergamot	4
„ caraway	2
„ cassia	2
„ lavender	8
„ cloves	1
„ petit-grain	1

Elder-flower Soap.

Oil of bergamot	8½
„ lavender	2
„ thyme	2
„ cloves	1
„ cassia	½
„ almonds	½

Glycerin Soap.

Oil of cassia	2
„ caraway	1
„ lavender	4
„ mirbane	1

Or—

Oil of lavender	4
„ bergamot	2½
„ thyme	1¼
„ cloves	1
„ caraway	¾

Or—

	oz.
Oil of rosemary	6
„ orange	3
„ cassia	1
„ thyme	1
„ mirbane	1

Honey Soap.

Oil of citronella	8
„ lemon-grass	2

Or—

Oil of citronella	8
„ lemon-grass	4
„ cassia	2

Lemon Soap.

Oil of lemon	8½
„ bergamot	4
„ lemon-grass	4½
„ cloves	2

Marsh-mallow Soap.

Oil of lavender	6
„ lemon-grass	4
„ peppermint	½
„ petit-grain	½

Millefleur Soap.

Oil of orange (Portugal)	10½
„ lavender	5
„ cloves	2½
„ nutmegs	5
Tincture of musk	5

N.B. Impregnate the fats used in this with vanilla, ambergris and rose-leaf.

Orange Soap.

	oz.
Oil of orange peel	8½
„ cinnamon	½
„ thyme	2

Rose Soap.

Oil of rose geranium	4
„ bergamot	2
„ rose	1
„ cinnamon	1

Or—

	oz.
Oil of rose..	2½
„ rose-geranium	2¼
„ cinnamon	1
„ bergamot	1¼
Tincture of ambergris	½

Violet Soap.

Powdered orris root..	71½
Oil of lemon	5
„ rhodium..	2½
„ thyme	2½
Tincture of musk	5

In connection with "Brown Windsor" soap, it may be mentioned that the more it is melted, cooled, manipulated, and remelted, the better it becomes, and that the scraps of various sorts of soap that accumulate in the factory are usually worked into this soap.

The intermediate quality of toilet soap is often made by the cold process (pp. 147–8), from the purest materials that can be prepared, and when the fatty matter (tallow, lard, &c., with usually a large percentage of coconut oil) and lye have been well stirred together, the colouring matter, perfume, &c., are added, and the whole is left quiet to effect the saponification. As previously directed, the fat should not exceed 120° F. (49° C.), and ½ its weight of caustic soda lye at 66½° Tw. (36° B.), should be stirred into it; in about 5 hours, when saponification occurs, the temperature will rise to 180° F. (82·2° C.). This method enables more delicate perfumes to be used, since they are added at so low a temperature. A marbled appearance may readily be given to this soap by drawing, in wavy lines, through the mixed fatty matter and lye, a steel blade dipped in colour ground up with oil; to produce a good effect, the peculiar wrist-turn should be used with the blade, such as is required to wield a fencing-foil well. It is obvious that these soaps retain their own natural glycerin, which is almost their only advantage, for they are exceedingly liable to contain both unsaponified fat and, what is far worse, free caustic alkali. This may arise from the saponification process not being complete, or

from errors on the part of the workman in adjusting the quantities. Moreover, the presence of a large quantity of cocoanut oil is decidedly objectionable.

The finest qualities of toilet soaps, however, require a great deal of manipulation by costly machinery, which has been chiefly devised by the French, although the Americans, with their well-known mechanical ingenuity, have recently constructed equally good machines. The basis of these soaps, or "stock" as it is technically termed, is sometimes carefully made by the cold process, from the purest possible tallow, lard, &c., with little if any cocoanut oil, which, if used, should be the Cochin variety. It is better, however, for reasons above given, to employ a carefully "fitted" soap for "stock" purposes. All the colouring matter, perfume, and other ingredients, are incorporated with the soap under hydraulic pressure, at the ordinary atmospheric temperature; hence the most delicate essences can be employed, even those that are extracted in the cold from plants. The first operation is to "strip" the stock-soap, i. e. to cut it up into strips or shavings; this may be done with a plane by hand, or by a machine (Fig. 75), whose essential parts are a revolving wheel A, upon which are set 4 or 6 knives, and a hopper F to contain the bars. After stripping, the soap is frequently dried somewhat, and it is then passed through the mill several times, while the colour, perfume, &c., are here added to it. The mill, which is shown in Figs. 76 and 77, consists essentially of three cylindrical contiguous rollers B, by whose action the soaps, colour, perfume, &c., after repeatedly running through, are blended into a thick homogeneous paste. When this has been effected, the soap is ready for the final operation, known as "plotting" (from the French, *pelotage*), in which the paste is subjected to enormous pressure, sometimes 3000–4000 lb. a sq. in., to form it into

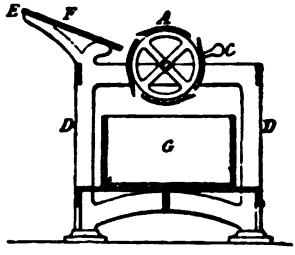
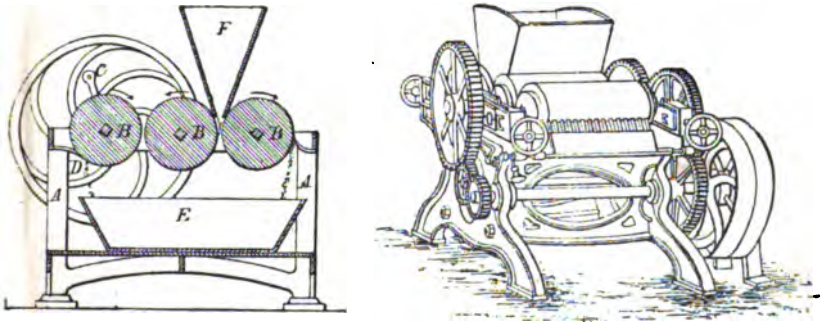


FIG. 75—STRIPPING MACHINE.

cakes, or into continuous bars from which cakes may be cut. Such a machine, known as Rutschman's hydraulic soap-plotting machine, made in Philadelphia, is shown in Fig. 78. It may be charged five times in a working day, and will "plot" 200 lb. at each operation. It is better to let each separate cake be "plotted" by this machine, but if bars are made, and the cakes are subsequently stamped, a powerful stamping-press must be employed, such as one of those described and figured on pp. 191–3. Cakes made in this way are not liable to crack in use, as those made by the other two processes are; before being packed they are not



FIGS. 76, 77.—SOAP MILLS.

unfrequently dried, and almost always polished. This may be done by hand with a cloth moistened with alcohol, or, according to Dupuis, by momentary exposure to a current of steam, which, if desired, may be previously passed through a cloth impregnated with any fragrant odour; it is said that no other method gives such a beautiful, even, and lustrous coating.

The whole process of manufacturing toilet soaps by this method was well illustrated at the International Health Exhibition of 1884, by Cleavers, in connection with their Terebene soap. Terebene is a volatile essential oil obtained by acting upon oil of turpentine with sulphuric acid, and

then distilling the product; it has a very agreeable smell, not unlike that of thyme.

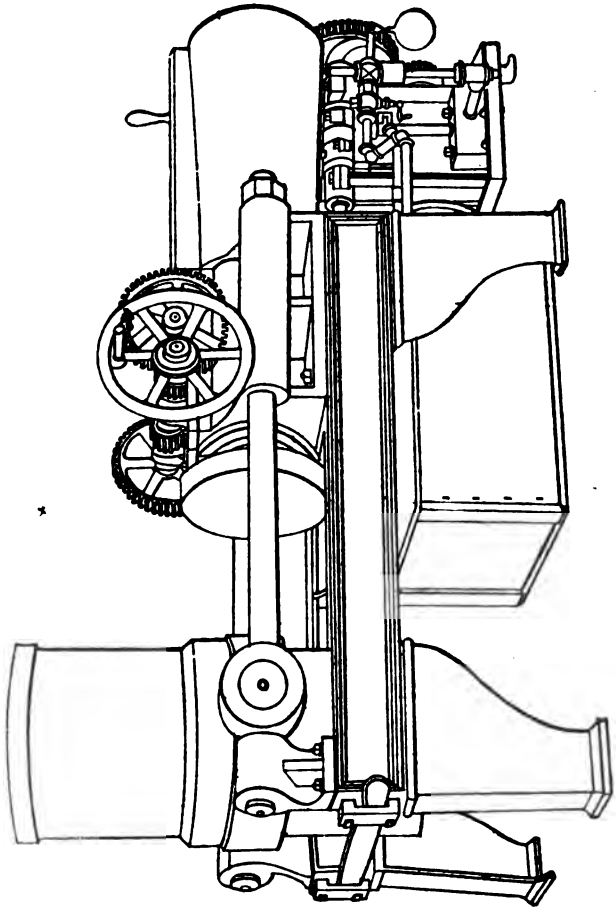


FIG. 78.—PLOTTING MACHINE.

To replace the long, laborious, and costly operations entailed in the present practice of making milled soaps, a Belgian firm, A. and E. Des Cressonières, have recently

introduced a continuous process, which does away with refrigerating apparatus and consequently also, the hardening into blocks, the cutting up of these, the shredding, the drying of the shreds, and the necessary apparatus for this purpose, as also the colouring and scenting while the soap is in a dry condition, and lastly the necessary crushing before entering the present apparatus. With the finer qualities, however, the scents of which might evaporate, the scenting of the soap is not effected till the end of the process, and may necessitate a supplementary kneading before the material is introduced into the presses.

The above results are obtained by causing the homogeneous mixture coming from the boiler to flow into a suitable receptacle situated in the vicinity, where the requisite colours and scents are added to the mass; and this is then made to flow between a series of hollow cylinders, open at the ends, between which the soap hardens as it proceeds, forming a thin sheet which is subjected alternately to cold and hot currents of air for removing the excessive moisture, and which passes eventually on to endless wire gauze travelling webs, on which the mass is subjected to further drying, and from which it is fed directly into presses.

The essential part of the new process consists in the employment of the apparatus for laminating and incorporating the material, and of a treatment with combined cold and hot air currents, the cold air serving only to render pasty and opaque the liquid soap, without depriving it of the necessary heat for its rapid desiccation, while the hot air retains the mass during the whole process at the required temperature for accomplishing this end.

The apparatus is illustrated in Fig. 79.

a is a shoot from the tank containing the liquid soap, situated above, which is charged from the boiler by means of a pump. The shoot is provided with a regulating valve *b*, and opens between small distributing cylinders *c*, revolving in contrary directions, towards each other. Against the lower sides of these cylinders bear guide plates *d*, which have

lips also bearing against the surface of the first cylinder *e* of the laminating apparatus.

This apparatus consists firstly of a series of cylinders arranged vertically above each other, and secondly, at the lower end of several other cylinders arranged horizontally, the framing being for this purpose of angular form, the vertical part being adapted to carry 4 vertical cylinders while the lower horizontal part carries 5. The cylinders might all be arranged vertically, but this would make the machine of inconvenient height.

The upper cylinders are not placed exactly in the same vertical axis, but so that their centres lie alternately slightly to the one and the other side of this axis, so that the points of contact of the cylinders also lie alternately to each side.

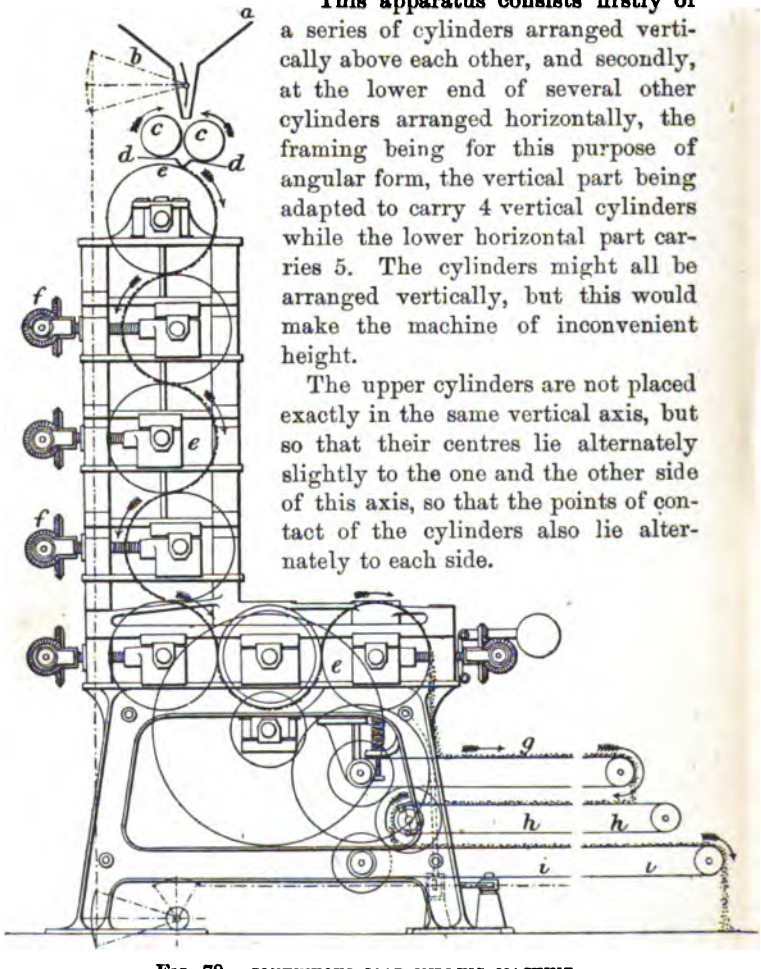


FIG. 79.—CONTINUOUS SOAP MILLING MACHINE.

They are consequently rendered adjustable by lateral displacement, and for this purpose the framing has horizontal

guides in which are arranged sliding bearings, which can be moved by means of screws actuated by bevel wheels *f*, which constitute nuts to the screws, and which are actuated simultaneously by two bevel pinions, fixed on one and the same shaft, carried in brackets on the framing, and rotated by a hand wheel. The same arrangement is employed for adjusting the horizontal cylinders.

The cylinders are rotated alternately in contrary directions at speeds which gradually increase for each successive cylinder, that is to say, if the upper cylinder, for example, makes 4 revolutions in a given time, the next one makes 5, the third 6, and so on, increasing one revolution for each cylinder. The cylinders are for this purpose each driven separately.

The driving shaft with fast and loose pulleys imparts motion by a pinion to the middle horizontal cylinder, and from this the motion is transmitted by gearing to the other cylinders.

In addition to the pinion, the driving shaft carries a toothed wheel corresponding to the wheel which drives the first endless wire gauze web *g*, from which the lower endless webs *h i* are driven by intermediate wheels.

In front of the last cylinder are two cutters, which serve to cut into flakes the sheets of laminated soap before arriving on the upper travelling web.

The apparatus works continuously, and the disengaging gear of the driving pulleys is connected to the regulating valve, so as to close this and stop the flow of soap when the machine is stopped.

The coloured and scented liquid mass descends in a thin sheet between the distributing cylinders *c*, from which it passes to the laminating cylinders *e*.

The machine being situated in a chamber the temperature of which can be regulated at will, and which may even be supplied with strata of air of different temperatures by means of fans, as may be required, the mass can be made to solidify into a sheet at any desired point of its transit.

The sheet is deprived of its moisture during its travel in passing through strata of air at the requisite temperature for

this purpose, and can thus be delivered from the last endless web in exactly the same condition as the soap delivered from the crushing rollers in the old method of treatment, but instead of requiring 8–12 days for arriving at this stage when treating about 6500 lb. of soap, the same quantity can be brought to the same condition in about 5–6 hours by this process.

In order still further to facilitate the desiccation, and also for preventing streakiness or want of uniformity in the coloration of the mass, the cylinders, or one or more of them, may have a longitudinal to-and-fro motion imparted to them, whereby the surface of the sheet of soap passing between them will be roughened or furrowed, thereby both breaking up any streaks, and facilitating the evaporation of the moisture. This movement may be imparted to the cylinders by means of cam grooves on their shafts, returning into themselves, and into which take fixed studs on the supports.

The process can be equally advantageously applied to the process of manufacture known as "remelters," as it enables this operation to be carried on without any loss, and preserves in the finished cakes the original appearance and form.

A writer in *Der Seifenfabrikant* remarks that while nearly all formulæ for violet soap agree that coconut oil and some tallow should be used, and that powdered orris root and the well-known characteristic perfumes, as well as tincture of musk, are to be employed, in almost everything else they differ.

One recommends that about 10 per cent. crude palm oil be used, another wants olive oil, and still others castor oil, and—to produce the well-known colour of this soap—sugar colouring or some other brown colour. Apart from the fact that the most worthless soaps in the market may often go by the name of violet soap, there are also some otherwise well-made brands which have one particular fault in common with even the worst make, viz. the colouring matter used produces stains.

Crude palm oil would not be a bad addition to the stock

in such soaps if it could be used as generally prescribed, viz. "fresh." But really fresh palm oil can rarely, if ever, be had; it is rancid on arrival. Even if it is in a passable condition, 10 per cent. would be too much, as it would cause almost indelible stains. From 3 to 5 per cent. would be all right.

While a cold-made violet soap cannot be compared with a well-made milled one, it is nevertheless well liked by many consumers. The problem then is how to make such without the addition of objectionable colouring matter. Olive oil in the stock is recommended by the writer mentioned, as well as castor oil, which has the property of fixing the colours, and helps to preserve the colour of crude palm oil. A small percentage of potash lye is recommended, and powdered orris root, powdered orange peel, liquid storax, and balsam of Peru will probably be of themselves sufficient to produce the necessary colour. If not, 3–4 per cent. of crude palm oil may be used in the stock, and a little vermilion (rubbed in oil) will do no harm.

A formula of this kind, which will give a very satisfactory result if carefully handled, is as follows:—50 lb. tallow (or 40 lb. tallow and 10 lb. olive oil), 40 lb. coconut oil, 15 lb. castor oil, 44 lb. soda lye, 71½° Tw. (38° B.), 6 lb. potash lye, 52½° Tw. (30° B.), 14 lb. orris root, powdered, 2 lb. orange peel, powdered, 2 lb. liquid storax.

The powdered orris root and orange peel are mixed with the oils and left therein for 10–12 hours, when the melted fats are also added. Next the storax is strained in through a cloth (and the vermilion, if wanted, is added). When everything is well mixed, and at a temperature of 100–110° F. (38°–43° C.) the lye is crutched in. When the soap is nearly ready for framing, it is perfumed with the following mixture:—

Oil of bergamot	350 parts
„ lavender	150 „
„ sassafras	20 „
Balsam of Peru	100 „
Musk (Tonquin)	3 „

The finished and hardened soap should remain well covered for at least a week before cutting it up. Soap cut too soon becomes covered by a white bloom, resembling the result of ordinary efflorescence.

A few hints on colour may now be given. Whenever it is desired to produce a mottled or marbled appearance in the soap, an insoluble colour must be employed; but whenever a uniform tint is required, preference should be given, whenever possible, to colours soluble in either water or alcohol, a condition fulfilled by numerous coal-tar colours. Care should be taken to choose those that are permanent, and unaffected by strong alkali. Salts of chromic acid should be avoided, since they are apt to turn green by transference of some of their oxygen to the fatty matter of the soap; the chromium borate, known as "Guignet's green," is very stable, and so are ultramarine and vermilion. The finest yellow is produced by infusion of saffron; red palm oil should be avoided for this purpose, as the soap bleaches by prolonged exposure to light. The resources of the dyer's art are constantly producing new tints, whose properties in relation to soap must be ascertained by that best of all tests, experiment.

With scented soaps, the first point to be observed is to select the proper shade of the flower corresponding with the perfume used; for instance, an almond soap is left white, rose soap is coloured pink or red, mignonette green, &c.

The colours from which the soapmaker may select are exceedingly numerous, for not only are most of the aniline colours adapted for his purpose, but also a very great number of mineral colours. Until a comparatively recent time the latter were probably exclusively employed, but the advance in the tar colour industry in later years has brought about a not inconsiderable change in this respect. A very prominent advantage of the mineral colours is their stability, i. e. not being changed or in any way affected on the exposure to light. This advantage, however, is offset in many cases by the wonderfully beautiful effect of numerous aniline colours, and by the more difficult method of application in the case of

the former. The specific gravity of mineral colours being rather high in most cases, they will naturally tend to settle towards the bottom, necessitating crutching of the soap until it is too thick to drop the colour. For mottled soap, however, cinnabar (vermilion) and ultramarine are still largely employed.

For transparent soap, of course, mineral colours are not applicable, as they would detract from their transparency; for milled soap, on the other hand, they are very well adapted, as also for cold-made soaps which require crutching anyway until a sufficient consistency is obtained to keep the colouring material suspended.

A notable disadvantage in the use of aniline colours, besides their sensitiveness to the action of light, is the fact that a majority of them are affected and partly destroyed by the action of alkali. A few of them are proof against a small excess of lye, and these may be used with good effect. Certain firms have made a specialty of manufacturing colours answering the peculiar requirements of soap and being very easy of application, as they are simply dissolved in boiling water and the solution stirred into the soap. To some colours a little weak lye is added; others are mixed with a little oil before they are added to the soap.

For a soluble red colour there were formerly used alkanet and cochineal; at present they have been displaced to a great extent, on account of their high cost, by "fuchsin," which is very cheap and of remarkable beauty. A very small amount of it suffices for an intense colour, nor is a large proportion desirable, as the soap would then stain. Very delicate tints are also produced by the phthalein colours, of which those named rosbengal, rhodamin, and eosin are most commonly used. These colours, when dissolved, have a green fluorescence, which heightens their beautiful effect.

There are also a number of the azo dyes which are suitable for soaps, and these, as well as the phthalein colours, are used principally for transparent soaps. For opaque soaps, both aniline and mineral reds are used, among the latter

being cinnabar, chrome red, and iron oxide. Chrome red is a basic chromate of lead which is now much used in place of vermilion, but as it becomes black on exposure to an atmosphere containing even traces only of sulphuretted hydrogen, it is not especially adapted for soap. Cinnabar gives a bright colour, but it is high in price. Iron oxide, known in the trade as colcothar, caput mortuum, &c., is only used for cheap soaps.

For yellow there are also a considerable number of colours. Among the natural colours there are prominent: saffron, orellin, curcumin (turmeric), and caramel (sugar colour); the first-named of these is now hardly used, owing to its high cost. Of the yellow aniline colours special mention is due to picric acid (trinitrophenol), martius yellow, naphthol yellow, the yellow azo dyes, and auramin. If it is an orange that is wanted, a trace of fuchsin (red) may be added to the yellow colours named. A mineral yellow is chrome yellow (chromate of lead), which has the same advantages and disadvantages as chrome red.

Transparent Soaps.—The peculiar appearance of these soaps is due to the use of alcohol in their fabrication, and it may be applied in two ways. Pure soaps—i. e. the soda compounds of the fatty acids, are soluble in alcohol, whereas the saline and other impurities which accompany even the best manufactured soaps to a small extent, are for the most part insoluble. The soaps are well dried, and then dissolved in strong alcohol; as the insoluble impurities fall to the bottom, they can readily be separated from the alcoholic solution by subsidence. The latter is then placed in a distillation apparatus, and the alcohol is distilled off, condensed, and recovered for use in the next operation; the soap, still hot, is left behind as a treacly mass, which cools to a semi-transparent solid. The hot soap is poured into moulds and allowed to cool; it is then set aside for some months, during which it is exposed to a temperature of 95° F. (35° C.). By this treatment it passes from the muddy semi-opaque condition to one of perfect transparency, and is then stamped,

polished, and sent into the markets. This is the process by which the famous Pears' soap is manufactured.

Inferior transparent soaps, however, are usually made by the cold process, but, to ensure success, very great exactitude in the proportions of the materials used is necessary, as well as much experience and skill. The fatty matters employed are tallow, coconut oil, lard, castor oil, and olive oil, in varying proportions, but all of the purest quality. For 100 parts by weight of fatty matter, 45 parts caustic soda lye at 76½° Tw. (40° B.), and 50–55 parts of alcohol of 95° should be used. One-half the lye should be stirred into the melted fat, the temperature of the mixture not exceeding 120° F. (49° C.), and, when thoroughly incorporated, the remainder of the lye, mixed with the alcohol, should be added; saponification will take place rapidly, and the perfume should now be added, and the whole cooled very gradually in frames; 20 parts glycerin added to the above will make a good transparent glycerin soap; occasionally some clear syrup of white sugar is added also. These soaps are seldom coloured, but any colour used in them should be quite transparent; it will be noticed that they do not become quite transparent until they have been exposed to the air for some days. The following is an analysis of one of these products, where a serious defect of this method, the presence of free caustic alkali, is evident.

Fatty acids	49·31
Combined alkali	6·37
Uncombined alkali	1·80
Water	22·40
Sugar, glycerin, &c.	19·40
	99·28

Many recipes for transparent soaps fail because the soaps do not retain their transparency. It would seem from experience that the warm process is preferable to the cold. The following is recommended for practical use where a good soap is required without employing alcohol:—48 lb. of tallow, 42 lb. of coconut oil, 60 lb. of castor oil, 90 lb. of a 64° Tw.

(35° B.) lye are taken, and the fats are saponified by running in the lye. The mixture is then covered and the temperature is allowed to rise to about 200° F. (93° C.); 40 lb. of sugar are dissolved in 42 lb. of water and 10 lb. of glycerin are added, and the whole is brought to a temperature of about 175° F. (80° C.). When the soap begins to form a thin clear mass with only a very thin skin, 16 lb. of sal soda, after being finely powdered, is thoroughly crutched in. Should the amount of soda specified be insufficient to make the soap quite hard, another 2 or 3 lb. may be added. After cooling to 140° F. (60° C.), any desired colour, dissolved in hot water, is then crutched in till the temperature sinks to 130 F. (54° C.). If a heavy scum appears on the surface colouring, there is not enough water, and the soap will not come out clear; sufficient water to remedy this must be added. The soap is then ultimately framed.

It is well to note, in connection with the preparation of transparent soaps in general, that it is important the exact quantities of fats and oils be taken, in order to accomplish which it is best to weigh off the quantities after melting, and clarify the same.

Solidified Glycerin.—The preparation of this by Price's Candle Co. is a trade secret, but Morfit recommends the following method:—Heat to 310° F. (154½° C.) a mixture of 350 lb. hot-pressed fatty acids, 150 lb. white oleic acid, 200 lb. best rosin. To this add 135 lb. Jarrow 52° ash (p. 150) in 25 gal. boiling water. When the soap-paste is quite homogeneous, which should be in about an hour, add 250 lb. pure glycerin and stir well. If a sample be not transparent when cold, add glycerin until this is the case, controlling the amount of glycerin by testing 2 lb. samples of the soap with glycerin over a gas flame. This soap has the following composition:—Fatty acids, 34·0; rosin, 13·0; soda, 4·6; water, 15·4; glycerin, 33·0.

CHAPTER XI.

THEORY OF THE ACTION OF SOAP—ITS VALUATION AND ANALYSIS.—DISTRIBUTION AND POSITION OF THE TRADE.

ACTION OF SOAP.

THE mode in which soap facilitates the removal of dirt is by no means clearly understood, and probably depends upon a variety of causes, partly physical, partly chemical. Unquestionably much of its power is due to the alkali it contains, which unites with and renders soluble the grease that forms so large a portion of much of our dirt; but it can hardly be true, as is maintained by some, that the value of a soap depends solely upon its percentage of alkali, since, if that were so, solutions of sodium silicate, carbonate, or aluminate, containing the same percentage of soda as soap, ought to do as much work, which is notoriously not the case. Further, since the proportion of alkali in a soap varies inversely as the equivalent weight of its fatty acids, those soaps with fatty acids of the smallest equivalent weights (e. g. coconut oil) ought to be the most advantageous. Gräger, who advocates this view, gives the following table of anhydrous soaps.

	Equivalent Weight.	Quantity of soap necessary to do the same work as 100 tallow soap.
Oleic acid soap	3800·95	115·1
Palm oil soap	3558·85	108·7
Tallow soap	3300·95	100·0
Coconut oil soap	3065·45	92·8

Cold water is never in contact with an alkaline stearate or oleate (the soap of commerce therefore) without decomposing it; the neutral salt is resolved into alkali, which dissolves, and an acid salt, which is precipitated as insoluble. Hence soap even in the purest cold water produces turbidity, although, when treated with *warm* water, it dissolves entirely. The alkali thus liberated acts upon the acid substances present in the surface which is being washed, and removes them in the form of emulsions or soluble compounds. But, it may be asked, does the fatty acid contribute directly to the detergent action of soaps? The question is thus answered by C. F. Cross, in his lecture on Soap at the Health Exhibition, previously quoted:—

"We have seen that the property of being slowly decomposed by water ensures in the use of soda soaps a gradual supply of free alkali, and this supply being obviously regulated by the combination of the alkali with the fatty acid, we recognise herein an important indirect influence of the fatty acid upon the cleansing action of soaps. It is also evident that the fatty acids, which are continuously liberated from their combination with the alkali, will be brought into contact with the substance undergoing the cleansing operation; indeed when they are of an acid nature—and, in most cases, surfaces exposed to the air are more or less acid—the fatty acid will be liberated upon the surface as the result of the union between the alkali and the acids in question. The fatty acids brought into contact with the surfaces of organic substances will tend to render them soft and smooth, and thus to lessen their adhesive attraction for foreign substances. Still this is rather an indirect effect, and the alkali would appear to be the sole effective agent in the cleansing work. But there remains to consider that property which is directly the result of the presence of the fatty acid in combination with the alkali, viz. the *lathering* of soap solutions. To understand the import of this property we may consider its most interesting demonstration, which is afforded in the soap-bubble. It is only of late years

that the sublime and the trivial have found a meeting-place in this curious phenomenon. That which was one of the delights of our childhood—which to the poet became the type of the evanescent—to the material philosopher affords food for solid reflection, and has been made the subject of recondite discussions.* The course of blowing a soap-bubble is a greater and greater attenuation of a soapy film, and the limit of this attenuation, i. e. the size of the bubble, is determined by the strength of the film, which is, in its turn, determined by the cohesion of the ultimate particles of soap. Now this cohesion, brought to demonstration by means of the bubble, upon an isolated portion of the liquid, is clearly a property of the solution itself, and must exert an important influence in overcoming the adhesion of foreign matters to surfaces."

Considerable light has been thrown upon the manner of removal of dirt by soap, by the researches upon *pedesis* of the late Prof. W. Stanley Jevons, F.R.S., who has given this name to a microscopic phenomenon long known as the "Brownian movement" of small particles. When clay is stirred up with water, and the whole is allowed to stand, the water clears itself very slowly, and microscopic examination shows that this is due to a kind of molecular movement of infinitesimally small particles of the clay. To this movement, Prof. Jevons gave the name pedetic action (*vide* 'Quarterly Journal of Science' for April 1878, No. LVIII.), and he found that it was largely influenced by the addition of certain substances to the water containing clay in suspension. Soap and sodium silicate enormously increase this pedetic action, or movement of the particles (Report of the British Association for the Advancement of Science, 1878, p. 435), and from observations made by Prof. Jevons, it seems clear that the action of such substances in promoting this movement of extremely minute particles, is an important factor in the cleansing power of soap.

* "See the Presidential Address of Lord Rayleigh at the British Association, 1884. Also the lecture of Prof. Rücker, at the Royal Institution, London, March 20, 1885."

When soap is rubbed in use against the surface to be cleansed, it is obvious that its consistency is an important consideration, since a harder soap requires much labour to detach a sufficient quantity, while a softer soap rubs away rapidly. It has been already shown that, *ceteris paribus*, the softness of a soap depends upon how much potash it contains; but where soda only is the base, the question of the comparative solubilities of the soda salts of the fatty acids has to be considered. While sodium oleate is freely soluble in 10 parts of water, sodium stearate is scarcely affected thereby; or in other words, the salts of oleic acid are far more soluble than those of stearic. Hence the hardness of a soap depends, not merely upon the base used, but upon the relative quantities of stearic and oleic acids in its composition; this point will be again referred to in the analysis of soaps. Moreover, the quantity of water contained by a soap influences very materially, not merely its hardness, but its rate of solubility in water, and especially in warm water. The quantity of water in normal, "neat," or genuine, soaps, varies from 16 to 35 per cent., according to their kind. Curd soaps, and the old-fashioned mottled soaps (p. 176), are, however, almost the only household soaps occurring in commerce, in which the normal amount of water has not been increased by the addition of some weak saline solution (p. 199). In low-priced coconut oil soaps, the addition of water is carried to an enormous extent; the writer has repeatedly seen a good-*looking* hard white or yellow soap, which contained 70 per cent. of water.

The impurity of the water employed in washing with soap has a material influence upon its consumption. Rain-water, and next to it, river or lake-water, is the best, while spring water should be avoided if possible; all such water is more or less "hard," owing to the presence in it of salts of lime (chiefly the bicarbonate and sulphate), some of which may be removed by boiling, or, more completely, by the addition of sodium carbonate. When a soluble potash or soda soap comes into contact with lime salts in solution, mutual decom-

position occurs, resulting in the formation of insoluble lime soaps, which have no detergent action. Until all the lime has been thus removed, the soda soap refuses to cleanse, and hence much of it is wasted. It is obvious also that the presence of any acid in the water, or on the surfaces to be cleansed, will decompose soap, uniting with its alkali, and destroying its detergent power. When nothing but hard water can be procured, or when much grease has to be removed, no soap will be found so economical as the old-fashioned curd-mottled, entangled in the interstices of which are appreciable quantities of caustic soda lye. Since the hardness of water is for the most part caused by the presence of salts of magnesia and lime, the laboratory test of the hardness of water will need but little explanation. The reagent employed is a solution of 10 *grm.* of a pure fatty acid soap (Castile soap is recommended) in 1 *litre* of weak alcohol. This is checked and standardised by a solution of known strength of a pure lime salt. The operation consists in gradually adding the soap solution to the hard water, shaking after each addition, until a permanent lather is formed. From the volume of soap solution used, the degree of hardness may be calculated.* The number of "degrees of hardness" indicates that the water contains soap-destroying salts approximately equivalent to that number of gr. of calcium carbonate per gal. of the water. The water supply of London is derived almost entirely from the rivers Thames and Lea, and its average hardness is 15°. This represents a destruction of soap equal to about 2 lb. for every 100 gal. of such water used for laundry purposes.

In many cases, some of the lime salts are kept in solution in water by the carbonic acid which all natural waters contain. When such water is heated, the carbonic acid is expelled, and the lime salts are deposited in an insoluble form, such as the "furring" in a tea-kettle or boiler; this is

* The lathering point may be very accurately determined by a piece of apparatus introduced by G. Loges (Chem. Zeit., 1884, p. 69) and figured in Journ. Soc. Chem. Ind., iii. (1884) 652.

why boiling lessens the hardness of water. The hardness is conveniently reduced on a small scale by the addition of sodium carbonate, or washing soda; on a large scale, some form of Clark's process may be adopted with advantage.*

There are few things which are so ill understood in practical life as the *real value*, or, what is the same thing, the proper price, of soap. From what has been said, it appears certain that the real value depends upon the amount of dry (anhydrous) soap present, and upon the proportion of stearic and palmitic acids to oleic, and of these three to that of the cocinic, laurostearic, &c. In other words, the determination of the following elements is necessary to arrive at an estimate of the value of a soap:—(1) The percentage of water; (2) the percentage of soda available for detergent purposes, (*a*) combined with the fatty acids, (*b*) as caustic, carbonate, silicate, or aluminate; (3) the percentage of fatty acids; (4) the melting, or rather the solidifying point (p. 100) of those fatty acids. To these may well be added a fifth, viz. some notion of the probable source whence they were derived, or some tests by which the more characteristic of them may be recognised; some hints in this direction will be found in Chap. IV.

It would be very greatly to the advantage of all large consumers of soap, as well as to soap manufacturers themselves, if soaps were to be sold, guaranteed to contain so much per cent. of fatty matter of a given melting-point, combined with the full quantity of soda necessary for its complete saponification, just as many other commercial products are sold at a price varying with the "test" of the quantity of the valuable constituent. It cannot be too widely known among consumers of all classes that those soaps are truly the most econoimal in which the percentage of water is lowest; but, on the other hand, it is unfortunately too often considered by the seller to be for his (immediate) interest to sell a soap which contains as large a quantity of

* 'Journal of the Society of Chemical Industry,' Feb. 29, 1884.

water as can be incorporated therein, consistently with the "saleable" character of the soap. What will "sell," not what will "wash" (in the metaphorical as well as in the literal sense of that word), is unfortunately the criterion too often adopted in the soap trade as in others. On this question of the ethics of the trade, we may quote again from Cross's lecture at the International Health Exhibition of 1884:—

"The love of gain, when indulged at the expense of others, is a vice which leads men to forsake art and take to artifice. In soap-making it takes the form of perverting ingenuity to the production of highly 'watered' soaps, the artifice consisting in keeping up the appearances of the soap in opposition to the influence of the water, which is in the direction of causing diminished hardness and loss of the 'graining' or 'strike' peculiar to normal soaps. . . . It is scarcely necessary to point out that the severe test of the wash-tub soon reveals the true character of these fraudulent soaps, their lack of the genuine substance causing them to 'waste.' In view of these facts, of which some of my audience will probably have been in ignorance, there should be no excuse for continuing to pay for water at the rate of a penny a pound or more."

In concluding these remarks upon this very important constituent of commercial soaps, and upon one of the unhappy results of keen competition, it may be well to point out—firstly, that in the case of the larger English firms, the manufacturers' price for a soap depends entirely upon the percentage of fatty acids, and the kinds of fats employed to produce them; secondly, that fraud consists in inducing a purchaser to believe that a given soap is more nearly a "neat" or genuine unsophisticated soap than it really is; and thirdly, that much of the evil complained of is due to the want of knowledge among purchasing consumers, who frequently insist upon some (adulterated) article being sold to them retail at actually a less price than the genuine article

would fetch in the wholesale market.* It follows, therefore, that it is very much in the power of consumers to assist the manufacturers in this important question.

VALUATION AND ANALYSIS OF SOAP.

Brief instructions will now be given for the most suitable methods, consistent with accuracy, for the analysis of soap; for fuller information, manuals of technical and of general chemical analysis should be consulted.†

Uniformity of Sample.—Great care should be taken to ensure this; since soap loses water rapidly on exposure, the soap should be sliced up in thin pieces, well shaken, and kept in a well-stoppered bottle. Other convenient plans are—(1) to weigh out at once, while the soap is fresh, all portions required for analysis; (2) to make a standard solution of the soap, say 100 *grm.* in 1 *lit.*, and measure off what is required, taking care to avoid loss by evaporation. In analysing case-hardened soaps (p. 188), care must be taken to see that the section of the bar includes a proper proportion of skin; sometimes separate analyses have to be made of different parts of a bar of these soaps.

Percentage of Water.—About 2 *grm.* of the soap is exposed in a wide-mouthed flask of about 100 *c.c.* capacity, to a temperature not exceeding 300° F. (149° C.), in an air- or oil-bath for 1 hour, and the loss in weight is noted. The flask should be weighed as soon as it is cool, and, where great accuracy is required, should be cooled under a bell-glass in presence of strong oil of vitriol, as anhydrous soap is very hygroscopic. The time required for the operation may be shortened by one-half, if a few drops of alcohol be added as soon as the soap has melted; the addition of a known weight of fine dry sand prevents the soap from swelling up too much.

* A very good instance of this is afforded by cocoa. A mixture of cocoa, sugar, starch, &c., is sold by retail grocers at a much lower figure than the wholesale price of even low qualities of *pure* cocoa.

† Allen's 'Commercial Organic Analysis' is one of the best books of its class.

No well-made soap should turn brown or be discoloured at this temperature.

Percentage of Soda (or Potash).—A burette is provided, divided into fifths of a *c.c.*, and a standard solution of acid, such as is directed in works on alkalimetry; either sulphuric or oxalic acid may be used. To determine the *total* percentage of soda present, dissolve 5 *grm.* of the soap in boiling water, and add to it the standard acid solution, stirring and boiling the whole time, until a permanent froth is no longer visible; from the number of *c.c.* of acid used, the amount of soda is readily calculated. Instead of the disappearance of froth, one of the numerous "indicators" may be used, as litmus, turmeric, eosin, &c. Another modification of the test is to add a decided excess of standard acid, boil the whole, and titrate back with a standard alkaline solution.

The amount of alkali combined with the fatty acids is probably most accurately determined thus. Dissolve the soap in alcohol; pass a current of carbonic acid gas through the solution, to convert into carbonate any caustic alkali present, filter off all insoluble matters, and determine the alkali in the filtrate. A simpler but less accurate method is to dissolve the soap in water, precipitate it thence by salt (repeating the operation a second time, to wash out all traces of uncombined alkali), and determine the alkali (A) in the soap only, (B) in the brine solution; A + B should, of course, be equal to the total percentage described above, while B alone gives the alkali present as free caustic, carbonate, silicate, borate, aluminate, &c. Errors, however, may arise from two sources:—(*a*) the impurities in the salts employed react on the soap; (*b*) partial decomposition of the soap by solution in water and re-precipitation.

If the alkali present be entirely potash, or entirely soda, the volumetric methods can be relied on; but if both are present, their proportions may be ascertained (1) by converting all the alkali present into oxalate, then evaporating to dryness and igniting the residue, which yields a mixture of carbonates in which the relative amounts of potash and

soda may be determined by the principles of indirect analysis; (2) by the direct determination of the potash with platinum tetrachloride.

Alkaline Salts.—The determination of soluble silica and alumina (as sodium silicate and aluminate), of sulphuric acid (as sodium sulphate), of chlorine (as common salt), and of other mineral constituents of soap, must be made in the acid solution that remains after decomposing the soap with a suitable mineral acid; for example, for sodium chloride, decompose with nitric acid, and titrate with silver nitrate—for silica, alumina, and sulphuric acid, decompose with hydrochloric acid, evaporate to dryness, filter off and weigh the insoluble silica, and determine the alumina, and the sulphuric acid by barium chloride, in the filtrate, by the usual methods. The most difficult problem is the carbonic acid, which is usually only present in very small quantity. It must be estimated gravimetrically (p. 132), and this may be done in the insoluble residue obtained by dissolving the soap in alcohol, or by operating in a somewhat larger apparatus (to give room for the solution of the soap, which *must* be aqueous) on or about 10 or 15 *grm.* of the soap itself.

Substances Insoluble in Water.—In a properly-made unadulterated soap these should only consist of colouring matters and mottling, and should never exceed 1 per cent. To estimate their amount, dissolve a known weight in water, decant the clear liquid, collect the deposit on a tared filter, wash, dry, and weigh. Organic impurities may be estimated by igniting this residue, and weighing again, when only mineral impurities remain. Starch or farina is detected by iodine; mineral impurities, by the ordinary methods of mineral analysis. Steatite, a silicate of magnesia and alumina, is an occasional constituent of low-class soaps.

Glycerin.—A weighed portion of the soap is dissolved in water, and decomposed with slight excess of sulphuric acid; the fatty acids are removed, and the acidulated solution is evaporated to dryness, dried at 230° F. (110° C.), and treated with absolute alcohol; the alcoholic solution of glycerin is

separated by filtration into a tared flask, from which the alcohol itself is distilled off. The result is invariably too low, owing to the volatility of glycerin in presence of warm aqueous vapour. A volumetric method has been described by Dr. Muter * for the indirect estimation of glycerin in soap, and in the lye resulting from the saponification of fats. It is based on the assumption that the solvent action of a solution of potash on cupric hydrate is directly proportional to the amount of glycerin in the liquid.

Carbolic Acid, &c.—Lowe recommends the following process. Take 50 grain-measures of aqueous solution of caustic soda at 69° Tw. (37° B.), dilute to 1000 gr.-m.; in this, dissolve by heat 100 gr. of the soap, then add 1000 gr.-m. saturated solution of common salt. Filter off, and wash with brine, the soap thus precipitated; slightly acidify the filtrate and washings with hydrochloric acid, and add thereto enough bromine-water to make the liquid permanently yellow. Warm the liquid till the precipitate melts, then let it cool; remove, carefully dry, and weigh the resulting mass, of which 331 parts correspond to 94 parts of carbolic acid. If the inferior qualities of carbolic acid have been used, the precipitate, which is tribromocresol, $C_6H_3Br_3O$, forms a sticky mass, owing to the liquid nature of the cresylic acid it contains. For the determination of the various constituents of toilet and medicated soaps, such as thymol soap, terebene soap, &c., the literature of the technology of those substances should be consulted.

Unsaponified fat, Hydrocarbon oils, &c.—These are best determined by agitating an aqueous solution of the soap with ether, which dissolves them all, and then treating the mixture in a separator as described on p. 96. When the ether has been distilled off, and the total constituents of this class weighed, the detection and estimation of them separately may be carried out as described on pp. 101–11.

Percentage and Examination of Fatty Acids.—A known weight of the soap (10 or 20 *grm.*, if only the percentage is

* 'Analyst,' vi. 41.

required, 50 or 100 *grm.* if the nature of the fat is to be ascertained), is dissolved in hot water. If any portion refuses to dissolve, as will be the case if steatite, clay, or starch have been mixed with the soap, the solution must be filtered, either in a hot closet, or through a funnel surrounded by hot water; if the filter be previously weighed, the insoluble portion can be weighed upon it after being washed and dried at or above 212° F. (100° C.); to the clear soap solution, an excess of sulphuric or hydrochloric acid is added, and the whole is gently boiled until the fatty acids are clear and transparent, and all clots have disappeared. If there is reason to believe that the fatty acids will be fluid, or even soft, and greasy, at the ordinary temperature, and a fat percentage only is desired, a weighed quantity of white wax or stearic acid, previously deprived of water (see p. 84), should be added at this stage. When the cake of fatty acids is cold, the liquid beneath should be removed, and the cake remelted over fresh hot water, to remove all traces of salts and acids. When cold, it may be partially dried with damp blotting-paper, if it is solid enough not to give up any oleic acid to that absorbent; it should then be all carefully transferred to a tared capsule, gradually heated to (but not kept at) at least 260° F. (127° C.), to expel the last traces of mechanically mixed water, and then weighed, the weight of wax or stearic acid added being of course deducted. Every 100 parts of fatty acids so obtained represent 105–106 parts of pure neutral fat used to make the soap. In this condition the fatty acids are hydrates, and from every 100 parts, 3·5 parts must be deducted in stating the analytical results for water chemically combined with them, because anhydrous soap [dried at 300° F. (149° C.)] does not contain these elements of water.

Another method, which is especially applicable where the fatty acids will not solidify at the ordinary temperature, and it is also desired to examine their nature, is to decompose the soap in a separator (p. 96) and agitate the liquid with petroleum spirit. When the aqueous liquid has been run

off, and all traces of the petroleum solution have been transferred to a tared flask, the spirit is distilled off, and the fatty acids are dried and weighed.

Both these methods assume the complete insolubility of the fatty acids in water. For practical purposes, this assumption is sufficiently correct, except in the case of coconut and palm nut oils, and butter. Various complicated methods have been described for estimating these soluble fatty acids, useful in the hands of skilled analysts, but unreliable in inexperienced hands. The best plan for a soapmaker is to operate in the same vessels which he habitually uses for fatty acid estimations, and in them to make a blank experiment with a suitable weight of pure (say) coconut oil, saponifying it, decomposing the soap, and weighing the fatty acids resulting therefrom. Probably 100 parts of oil will give him about 93 parts of fatty acid. Using these data, the quantity of coconut oil used to make a given soap (which is what the soapmaker wants to know) can be readily calculated from the percentage of fatty acids obtained from it by analysis. The same proceeding applies, *mutatis mutandis*, to any fat.

To ascertain the nature of the fatty acids, the melting-, or solidifying-point of the mixture should first be taken by one of the methods given on pp. 99–101. Where the presence of coconut or palm nut oil is suspected, the question may be determined, and their quantity estimated with a close approach to accuracy, by the test described on p. 105. In any case, and more especially after the record of many such data, the determination of their specific gravity (p. 97), will afford much useful material for judgment as to the nature of the fatty acids, and it is especially valuable in the estimation of the amount of rosin present.

Another mode of attacking the same problem (proposed by Dalican, but due originally to F. Jean) is here given, with the remark that Sutherland's process, by which the rosin is oxidised by nitric acid, and Rampal's process, in which the rosin is precipitated in a finely-divided state by

throwing an alcoholic solution of the fatty acids into water, are both unreliable. Dissolve 10 *grm.* of the soap in 100 *grm.* water, add enough concentrated soda lye to precipitate the soap; some resinates remain in the liquor, which is neutralised, evaporated to dryness, and the rosin is extracted with alcohol which may be distilled off, and the rosin weighed (A). Then dissolve the precipitated soap in water, and add excess of barium chloride; collect and dry this baryta soap, and extract it with ether, which dissolves out only the resinates; evaporate off the ether, and treat the resinates with boiling water and sulphuric acid, which sets the rosin free; it may then, if necessary, be similarly dissolved out by alcohol, or may be merely collected on a weighed filter, and its weight (B) noted; A + B is the weight of rosin in the soap. The portion insoluble in ether may then be suspended in water, decomposed with sulphuric acid, and the fatty acids collected, dried, and weighed. Another tolerably simple process has been devised by Barfoed, with which some analysts have obtained very encouraging results.*

The method, however, which will probably supersede all others, is that described by T. S. Gladding in the 'Chemical News' for April 14, 1882. About 0·5 *grm.* of the mixture of fatty acids and rosin is dissolved in 20 *c.c.* of 95 per cent. alcohol, and thoroughly saponified with a slight excess of alcoholic potash. The solution is then mixed with enough ether to make it up to 100 *c.c.*, and to it is added 1 *grm.* of very finely powdered neutral silver nitrate, the whole being well shaken for 10 minutes. After the precipitate has subsided, an aliquot part of the solution is measured off, and decomposed with dilute hydrochloric acid (1 : 2); an aliquot part of the same ethereous solution remaining after this treatment, is evaporated in a tared dish, and weighed as rosin, a small deduction (0·00235 *grm.* for 10 *cc.*) being made for oleic acid.

Shorter, but less reliable, methods than the above have frequently been proposed for determining the value of soap.

* *Vide* Journ. Chem. Soc., xxix. 773, and Zeit. Anal. Chem., xiv. 20.

To shorten the operation of weighing the fatty acids, many methods have been tried for measuring them, by collecting them in a long-necked flask, graduated, or in a graduated tube attached thereto. Whenever this is done, the weight can only be arrived at from the estimated sp. gr. of the fatty acids, and as this is very variable, the method is at best an approximate one, though useful in the factory when that sp. gr. is known. Buchner decomposes 16·66 *grm.*, and measures the fatty acids to the $\frac{1}{5}$ *c.c.*, multiplying by 0·93 to get the weight in *grm.* He also gives the following useful table, on the basis that 100 lb. fat produce 155 lb. soap and about 6·25 lb. glycerin; the 3 last columns are of general use, when the "fatty acids per cent." are determined by weight.

No. of *c.c.* of fatty acids from 16⅔ *grm.* soap.	Sp. gr. of fatty acids.	Mean weight of the fatty acids in *grm.*	Fat used for 100 lb. soap.	"Neat" or grain soap in 155 lb. of soap examined.	100 parts of the soap contain of water, soda, glycerin, &c.	100 parts of the soap contain of real grain soap.
0·5	0·93	0·46	3·13	4·85	97	3
5	"	4·65	31·30	48·50	69	31
6	"	5·58	37·56	58·20	63	37
7	"	6·51	43·82	67·90	57	43
8	"	7·44	50·08	77·60	51	49
9	"	8·37	56·34	87·30	44	56
10	"	9·30	62·60	97·00	38	62
11	"	10·23	68·86	106·7	32	68
12	"	11·16	75·12	116·4	26	74
13	"	12·09	81·38	126·1	20	80
14	"	13·02	84·64	135·8	13	87
15	"	13·95	93·90	145·5	7	93

A short way of ascertaining whether there is much besides pure soap and water in a sample of soap, is to treat it with strong warm alcohol, which dissolves nothing but the soap, and excess of caustic soda, if any; this last may be removed by a stream of carbonic acid gas. The insoluble residue may be collected on a tared filter, washed with alcohol, dried, and weighed.

The Industrial Society of Mulhouse awarded a prize to

Dr. Leeds' Scheme for Soap Analysis.

Weigh out 5 grammes. Dry at 212° F. (100° C.). Loss corresponds to water.

Treat with petroleum ether.

Extract is uncombined fat. Dry at 230° F. (110° C.) and weigh.

Residue is soap and mineral constituents. Treat with alcohol.

Extract is soap (fatty anhydride, resin and combined alkali), glycerin and free alkali. Add two or three drops of phenol-phthalein. If necessary, titrate with normal sulphuric acid.

H_2SO_4 used corresponds to free alkali. Calculate as NaHO.

Add a large excess of water and boil off the alcohol. Decompose with excess of normal H_2SO_4. Boil, filter, and wash.

Filtrate.—Combined soda and glycerin. Titrate with normal soda solution.

H_2SO_4 used corresponds to combined soda in soap. Calculate as Na_2O.

After titration evaporate to dryness on the water-bath. Treat with absolute alcohol. Evaporate the alcoholic solution to dryness in a tared dish and weigh as glycerin.

Residue.—Fatty acids and resin. Dry at 230° F. (110° C.), and weigh. Dissolve an aliquot part in 20 *c.c.* strong alcohol, and, using phenol-phthalein as an indicator, saponify with soda in slight excess. Boil, cool, and add ether to 100 *c.c.* Decompose with $AgNO_3$ by adding in fine powder, and shake well for 10 minutes. Allow to settle.

Precipitate is stearate, palmitate, and oleate of silver.

Solution.—Resinate of silver. Filter 50 *c.c.* from the total 100 *c.c.* Decompose with 20 *c.c.* HCl (1 : 2). Allow the AgCl to settle, and evaporate an aliquot part of the ethereal solution in a tared dish. Dry at 230° F. (110° C.), and weigh. After applying correction for oleic acid, the weight corresponds to the rosin. This weight subtracted from the combined weight of fatty acid and rosin gives the fatty acids.

Residue.—Na_2CO_3, NaCl, Na_2SO_4, sodium silicate, starch and insoluble residue. Wash with 60 *c.c.* water.

Filtrate.—Na_2CO_3, NaCl, Na_2SO_4 and sodium silicate. Divide into four equal parts.

Na_2CO_3. Titrate with normal H_2SO_4, and calculate as Na_2CO_3.

NaCl. Titrate with $AgNO_3$ or weigh as AgCl. Calculate as NaCl.

Na_2SO_4. Weigh as $BaSO_4$. Calculate to Na_2SO_4.

Sodium silicate. Decompose with HCl and determine soda combined in silicate and silica.

Residue.—Starch and insoluble residue. Dry the filter and weigh. The weight is the starch and insoluble residue. Starch.—Convert the starch into $C_6H_{12}O_6$. Titrate with Fehling's solution. Subtract the weight of starch found, and the difference is the insoluble mineral constituents.

Cailletet for a method of analysing soap without more weighings than that of the soap itself. Minute instructions are given in the Bulletin of the Society, No. 144, tome xxix. p. 8. Suffice it to say here that, in the first place, much information may be gained for industrial purposes by attentively observing the behaviour of the soap with hot and cold water; 10 *grm.* of the soap are then decomposed by excess of standard acid in presence of a measured volume of turpentine oil, the increase in volume of which, multiplied by the sp. gr. of the fatty acids, gives their weight. The acid solution is titrated back with soda, and the soda per cent. is calculated. It is, however, stated erroneously, that the turpentine does not dissolve the rosin, and that thus the presence of rosin may be detected, and even estimated.

A good scheme for the complete analysis of soap, first published in the 'Chemical News' by Prof. Albert R. Leeds, is reproduced on the opposite page.

It is somewhat remarkable that there are very few published analyses of soaps upon which much reliance can be placed, the amount of information afforded by them being very meagre, and usually confined to the percentage of fatty acids and of the "total soda," the remainder being entered as "Water, &c." A valuable table is given by Allen (*loc. cit.* p. 255), and appended are some published by the author in 1881, with a few additions. It must be remembered that the fatty acids are weighed as hydrates (pp. 9, 11), and hence the sum of the constituents should always exceed 100 per cent. Approximately, 100 parts fatty acids as weighed represent 97 parts of the "anhydrides," as they exist, for example, in soap dried at 300° F. (149° C.).*

* See also, H. T. Vulté, "Soap: its Manufacture and Analysis," 'School of Mines Quarterly,' N.Y., xxiii. 293.

A good Yellow Soap known as "Primrose" in South and West of England.

	Per cent.
Water	32·8
Soda, total	6·7
Sodium chloride	0·2
Fatty acids	62·3
	102·0

An old-fashioned Grease Mottled Soap.

	Per cent.
Water	29·8
Soda with fat	7·0
„ (free caustic)	0·6
Sodium chloride	0·1
Fatty acids	64·7
	102·2

A genuine "Cold-water" Soap.

	Per cent.
Water	22·0
Soda with fat	7·3
„ with silica, &c.	1·8*
Silica	1·6
Sodium chloride and sulphate	0·4
Fatty acids	70·2
	103·3

* Or 5 per cent. sodium silicate sp. gr. 1·700.

Manufacturers' Brown Oil Soap, from Oleic Acid.

	Per cent.
Water	21·00
Combined soda	7·88
Free soda	1·00
Other soda salts	1·00
Fatty acids	68·60
	99·48

A Blue (red or grey) Mottled Soap.

	Per cent.
Water	44·3
Soda with fat	5·2
„ (free, or) as silicate	0·8
Silica	1·3*
Sodium chloride	0·8
„ sulphate	0·3
Mottling and insoluble	0·7
Fatty acids	47·5
	100·9

* Equal to 3·9 per cent. sodium silicate at 1·700 sp. gr. Some of the "case-hardened" mottled soaps (p. 176) contain 6 or 7 per cent. of sodium chloride.

A neutral Curd Soap, for Manufacturers.

	Per cent.
Water	28·0
Soda with fat	7·0
„ free, &c.	0·0
Sodium chloride	0·2
Fatty acids	67·9
	103·1

Marine Soap, for emigrants' use (C. Hope).

	Per cent.
Fatty acids	20·02
Soda existing as soap	3·11
Silica	9·00
Soda as silicate	3·98
Sodium chloride	5·13
„ sulphate	0·35
„ carbonate	2·35
„ hydrate	0·65
Lime, iron oxide, &c.	0·16
Water	53·32
Glycerin (calcul.)	2·80
	100·87

Primrose Soap as in the North of England.

	Per cent.
Water	50·40
Combined soda	5·41
Soda, free and as silicate	1·21
Silicic acid	0·94
Sodium chloride	0·42
„ sulphate	0·13
Fatty acids	42·66
	101·17

THE SOAP TRADE.

A few remarks upon the location, prospects, legislative condition, and other general considerations connected with the soap trade, may fitly close these chapters on this important manufacture.

This industry is by no means localised in any one part of the British Islands; but, although the total amount of soap made in England is probably greater now than it ever was, the tendency of the last 35 years has been in the direction of concentrating the manufacture in the hands of a few large firms. Probably the oldest soapworks in the country are, or at any rate until recently were, to be found in Bristol, which still retains great reputation for its soap. A relic of this may be found at the present day in Holland, in some parts of which no soap can be sold which is not stamped with the word BRISTOL. Of the two soapworks now left in that city, the larger, belonging to the firm of Christopher Thomas & Bros., was established in 1745.

The abolition of the duty on soap in 1853, then about 2*d.* a lb., and producing a revenue of upwards of 1,000,000*l.*, naturally gave an immense impulse to improvements in the manufacture, and various valuable patents were very shortly taken out, the most important of which were those of W. Gossage for silicated soaps; that of T. Thomas for cheap detergent soaps made from mixtures of neat soap, with sodium silicate and sulphate; that of Blake & Maxwell for hydrated soaps (p. 169); and those of C. N. Kottula, for various improvements in the making of blue-mottled and other soaps. An association of soap-manufacturers in England holds quarterly meetings, at which prices are revised, common action is agreed upon, and legislative enactments affecting the trade are discussed and watched. All soapworks, in common with other factories, are subject to inspection by Factory Inspectors acting under the Government, and to the visits of a certified surgeon who regulates the admission to work of young persons under 18 years of age, and also per-

forms various functions of inspection and report in cases of accidents, &c. It is his duty to see that certain sanitary precautions are duly enforced.

In a well-managed soapworks, the sources of nuisance are very slight, and comparatively inoffensive; most of them arise, not from the actual process of soapmaking itself, but from the preliminary operations of refining, purifying, and bleaching the fatty matters employed (Chap. IV.). They may all be obviated by conducting such operations in closed vessels provided with trunks communicating with the draught of a flue; when impure and rancid fats are used direct for soapmaking, the copper should be provided with a cover and a similar trunk. In default of a flue-draught, a fan or a jet of steam may be used to create a good current of air. In some cases the obnoxious fumes may be conducted with advantage into the ashpit of a fireplace or furnace, where they are at once consumed; care must be taken, however, that they do not contain enough vapour of fatty matters to make them inflammable. In dealing with these matters, water-lutes, water-joints, wash-bottles, and similar contrivances will be found very useful, but it is an obvious precaution so to arrange the apparatus that there may be no chance of any water being "sucked back" (by a suddenly formed vacuum) into heated tallow or oils, else an explosion might ensue. Manufacturers will derive some useful hints upon the prevention of odorous nuisances in their works, from Dr. Ballard's reports to the Local Government Board.

Since the removal of the duty, there are few means of forming an estimate of the extent of the soap trade in England; it is known, however, that many of the larger houses make much more than 5000 tons a year, while a few make over 10,000, and it is stated that one house is capable of turning out 500 tons in a week when necessary. The total annual production in the United Kingdom was estimated at 250,000 tons by Prof. Roscoe, in his inaugural address as President of the Society of Chemical Industry, in June 1881.

In France, the chief seat of the industry is at Marseilles, while a not inconsiderable amount of common soap, and nearly all the toilet soaps, are made in Paris. In a report on the exhibits at Paris in 1878, it was stated that the French soap trade had been for some time stationary at about 220,000 tons per annum, but was then declining, owing to practices not very creditable to the manufacturers.

In Germany, and other parts of the Continent, soft soaps are much more proportionately in vogue for laundry and other purposes than in England, while the chief hard soaps made are for toilet purposes.

In the United States, Kirk & Co. of Chicago have probably the largest trade, but they are closely approached by Babbitt & Co., and Colgate & Son, of New York. The changes which have lately passed over the trade in America have been already alluded to (pp. 198–9). It may be said, without fear of contradiction, that while perhaps for fancy toilet soaps the palm must be given to France, England and the United States are pre-eminently the countries where the manufacture of the different varieties of household and factory soaps is most clearly understood, and carried out on the largest scale, and in the best manner.

In Australasia, the largest soapworks are those belonging to J. Kitchen & Sons, and Apollo Co., Limited. The chief seat of the trade is in Melbourne; and the products shown at the Melbourne International Exhibition compared very favourably with similar articles manufactured in England. As in America, nearly the whole of the alkali used is imported, chiefly from England.

CHAPTER XII.

LUBRICANTS.*

THE purpose of lubrication is the reduction of friction between contiguous moving surfaces, and this is usually achieved by the introduction of some medium which may be, in its normal condition, either liquid, as oil, or solid, as tallow or grease. By common consent oils appear to be regarded as at once the most convenient and most efficient lubricants, and have to a large extent, notably in the case of railway carriage and waggon axles, superseded greases where these were formerly used.

Until the fifth or sixth decade of the present century the principal oils available for lubrication were sperm, neatsfoot, lard, olive, refined rape, and castor. For textile and other kinds of light machinery, sperm oil was commonly used. For engines, shafting and heavier machinery, neatsfoot, lard, olive or refined rape oil was preferred. For marine engines, refined rape, olive, lard, and, in the case of vessels trading in hot climates, castor oils, were regarded as standard lubricants.

Until a somewhat later period, beef or mutton tallow or suet was almost universally used for the lubrication of steam cylinders and valves, and occasionally for the lubrication of heavy bearings and hot necks, while for the lubrication of railway carriage and wagon axles, palm grease was almost exclusively used.

In 1850 Mr. James Young obtained his famous patent for the production of hydro-carbon oils by the distillation of coal at a low red heat, which was destined to revolutionise

* The article on Lubricants has been written by Mr. J. Veitch Wilson specially for this work.

the trade in lubricating as well as in illuminating oils. But although, despite the inferior quality of the new as compared with the older lubricants, Mr. Young succeeded in finding a market for mineral oils, their use was limited to the lighter kinds of machinery, and even for this purpose it was necessary to enrich them by the addition of liberal percentages of animal or vegetable oil. The name "Finished Liquor," originally applied to the heavier mineral oils by Mr. Young, appears to indicate that even he did not regard them as entitled in their natural state to be described as "oils," and the writer has been informed by some of the earliest users of mineral oils that they were induced to adopt them rather on account of the protection which their disagreeable taste and smell afforded against the purloining proclivities of their operatives, than for any practical or economic advantages which they offered. The expiry of Young's patent in 1864 opened the field for the energies of a host of investigators and manufacturers, who speedily, in respect to mineral lubricating oils, surpassed the best results that had been obtained under Young's patent, and greatly extended the production and the use of these oils.

In 1859, petroleum, which had previously been collected in various districts of America in small quantities from natural springs, or from the surfaces of creeks or streams, and had been used mainly for medicinal purposes, was obtained in large quantities by boring in some of the Western States, and an apparently inexhaustible supply of illuminating and lubricating oils was placed at the disposal of the world.

Russian petroleum has a much older history than either its American rival or than Scotch paraffin oil, for, while tradition points to the knowledge, as early as the fifth or sixth century before the Christian era, of mysterious springs and fires which occurred in Western Asia, where the civilisation of these days had its seat, and which were regarded with superstitious reverence by the surrounding nations or tribes, we have authentic records that the value of the oils which

were found exuding from the earth in the neighbourhood of Baku on the Caspian Sea had been recognised in the days of Peter the Great (circa 1700), and that they were then collected and used in their natural condition for lighting and heating as well as for medicinal purposes. The importance of the trade in these oils gave rise to a series of struggles between Russia and Persia for possession of the district in which they were produced, and, in accordance with the fortunes of war, it changed hands several times during the eighteenth century. In 1801 the peninsula of Apsheron in which Baku is situated was finally reannexed by Russia, and from that date the oil trade has steadily increased. The late Charles Marvin says that in 1813 the monopoly for the extraction of petroleum was farmed out to a merchant named Meerzoeff, and continued in force till 1872. In 1819 the yield of petroleum was estimated at 4000 tons. From 1832 till 1860 the average annual production was, according to Redwood, about 5000 tons, and in 1870 it had risen to 27,000 tons. It was not, however, until the development of the Scotch and American trade in mineral oils, and the discovery of processes for the production of useful illuminants and lubricants from these, that the value of Russian petroleum was fully appreciated and attention was directed to the development of the industry. This was greatly promoted by the abolition of the monopoly in 1872, the immediate result of which was that native and foreign capital, energy and experience were devoted to the exploitation of the Russian oil fields by systematic drilling for the raw material, by the erection of refineries on approved principles, and by improvements in the means of transport. Foremost among the pioneers in this new departure we find the name of Nobel Brothers, two Swedish engineers, who in 1875 turned their attention to the production and refining of illuminating oils, and to the distribution of these throughout the Russian Empire and Europe.

About the same time V. I. Ragosine, a Russian, devoted himself to the more difficult task of producing, from Russian materials, a series of pale and dark lubricating oils which

should meet the requirements of European buyers, and it is within the writer's knowledge that this gentleman, to whom pertains the credit of being the father of the Russian lubricating oil trade, had, within a few years from the abolition of the monopoly, not only succeeded in producing good merchantable oils, but had established branches in, and had introduced his products to, the chief manufacturing countries of Western Europe.

Under such fostering influences the production rapidly increased. In 1880 it had risen to 400,000 tons, and in 1890 to 3,850,000 tons per annum, at which latter date it may be interesting to note that the total American annual production was estimated at 34,820,306 barrels of 42 American gallons * each, or say about 4,500,000 tons.

From this brief account of the introduction of mineral oils, it will be seen that the animal and vegetable oils which formerly constituted the sole materials available for lubrication were supplemented by the addition successively of oils from shale and from American and Russian petroleum, and, when account is taken of the enormous accessions which, of late years, have been made to the mechanical power of the world and to the severe conditions of speed, pressure, and temperature by which these extensions are accompanied, it may be doubted whether, but for the timely introduction of mineral oils, the supplies of the older lubricants would have been equal to the demands made upon them.

The general appearance, characteristics, and origin of the principal animal and vegetable oils are so well known as to render any discussion of them unnecessary, but mineral oils call for further consideration. Mineral lubricating oils may be divided into three well-defined groups, viz.:—

(*a*) Transparent oils, pale to dark amber in colour, produced by distillation and subsequent washing with sulphuric acid and soda, either from crude petroleum or shale oil, or from the residuum obtained in the production of burning oils.

(*b*) Opaque oils of dark green colour, prepared by settling

* The American gallon is the old English wine gallon—one-sixth less than the British imperial gallon.

crude petroleum in tanks, and by filtration through canvas, wire gauze, cotton waste, sawdust, or other mechanical medium, in order to remove sand or grit, and by partial distillation to remove the lighter and more volatile fractions.

(*c*) Oils of the vaseline type, produced by filtering oils of the foregoing class (*b*) through animal charcoal, by which the natural bitumen or tar is removed, and a pure petroleum jelly is obtained.

In the earlier days of the mineral oil trade, the oils of group "*a*," commonly referred to as "refined oils," were often low in flashing point, disagreeable in smell, and deficient in body, and were only rendered available for lubrication by liberal additions of fatty oils. American refined lubricating oils—to which Americans apply the name "paraffin oils" as denoting the connection of these oils with the paraffin-wax yielding section of the distillate—evinced a distinct advance in all three respects. Still further advantages were obtained by the introduction of Russian refined oils which, in addition to body or viscosity much greater than that of any other mineral oils, or of any animal or vegetable oil, except castor, are remarkable for their very low freezing point (due to the entire absence of paraffin in their composition), and are free from the tendency, present in many American oils, to produce varnish-like films upon, and to discolour or stain bright metal. These successive improvements had the effect of greatly extending the use of mineral oils by rendering them available for many kinds of machinery and for purposes for which they had formerly been found unsuitable, by permitting of large reductions in the fatty matter usually added to them, and, in some cases, by dispensing with it altogether. (See Table A, on p. 263.)

The oils of the second group (*b*) may be subdivided into two classes: 1st, those in which the operation of "reducing," or concentrating, by the removal of the lighter fractions by distillation, has been arrested at the point necessary to produce oils of sufficient body or viscosity for use on ordinary bearings and at ordinary temperatures; these oils, which are of various

TABLE A.

Refined (Pale) Mineral Oils.

Name of Oil.	Sp. Gr. at 60° Fahr.	Viscosity.*			Flashing Point. Close Test. Fahr.	Setting Point, about. Fahr.	Fatty Acid.
		At 70° F.	At 120° F.	At 180° F.			
Scotch, nominal, 890	890–895	100–130	40–50	..	320–350	32	Nil.
" " 885	885–890	75–100	35–40	..	300–325	32	"
" " 875	875–880	50–60	25–30	..	300–325	32	"
American " 915	915–920	400–425	90–100	35–40	375–425	32	"
" " 905	905–910	200–225	55–65	..	350–400	32	"
" " 885	885–890	75–100	25–40	..	325–350	32	"
" " 875	875–880	65–75	30–35	..	325–350	32	"
Russian, heavy red, ...	910–915	1200–1500	200–250	50–70	400–425	25	"
" " 910	905–912	700–800	125–150	45–50	350–375	25	"
" " 895	895–900	220–250	60–65	..	325–350	15	"
" " 895	895–900	125–175	..	..	300–325	10	"

* Standard for viscosity, sperm oil at 70° F. = 100.

degrees of consistency, flashing point and setting point, are largely used, with or without the addition of a little animal or vegetable oil, for the lubrication of railway carriage and wagon axles, for pulleys and axles in underground workings, and by engineers and others to whom their dark colour is not likely to cause trouble. 2nd, oils in which the operation of "reducing" has been continued until the body and the flash point have been raised so high as to render them suitable for the lubrication of cylinders and valves, or other bearings working at high temperatures. (See Tables B and C, p. 265.)

The third group (*c*) comprises a series of dark amber-coloured and semi-transparent oils, produced from oils of group "*b*" by further filtration at high temperatures through animal charcoal, by which the natural bituminous matters are removed, the colour improved, and the distributive properties of the oil in contact with steam promoted and extended. (See Table C, on p. 265.)

Much difference of opinion exists regarding the relative merits of "natural" and of "charcoal filtered" cylinder oils, and in practice consumers appear to be pretty equally divided in favour of the two classes. When, however, it is borne in mind that "natural" cylinder oils yield, on complete combustion, 2½ per cent. to 4 per cent. of dry coke, which, when dissolved in the oil must represent a much larger percentage of tarry matter, while the better qualities of "charcoal filtered" oils yield by similar treatment only a trace of coke, it must be evident that where, from the construction of the engine, or from other conditions, tarry deposits in cylinders, condensers, or boilers are to be apprehended and avoided, preference ought to be given to charcoal filtered oils.

The properties which affect the value of oils as lubricants may be conveniently considered under two heads, viz. :—

Negative, or those which prohibit the use of certain oils.

Positive, or those which (a minimum standard being required for safety), by variation within permitted limits, determine the suitability of oils for different purposes.

TABLE B.—Natural (Dark) Mineral Oils for Machinery.

Name of Oil.	Sp. Gr. at 60° F.	Viscosity.*			Flashing Point. Close Test. Fahr.	Setting Point, about. Fahr.	Fatty Acid.
		At 70° F.	At 120° F.	At 180° F.			
American summer dark	890–895	..	250–300	70–75	400–425	40–50	Nil.
„ medium	880–885	550–700	110–125	40–50	350–400	25–30	„
„ winter	880–885	350–400	90–100	35–40	325–375	25–30	„
Russian residuum	910–915	750–1000	150–200	45–60	250–300	25–30	„

* Standard for viscosity, sperm oil at 70° F. = 100.

TABLE C.—Natural and Filtered Mineral Oils for Cylinders.

Name of Oil.	Sp. Gr. at 60° F.	Viscosity.*			Flashing Point. Close Test. Fahr.	Setting Point, about. Fahr.	Fatty Acid.
		At 70° F.	At 120° F.	At 180° F.			
American heavy dark	900–905	..	1750–2000	350–400	500–550	40–45	Nil.
„ extra „	900–905	..	2000–2500	400–450	525 575	35–40	„
„ medium „	895–900	..	1200–1400	300–350	500–525	40–45	„
„ heavy filtered ..	890–895	..	1400–1500	300–350	500–550	60–70	„
„ medium „	890–895	..	1000–1200	250–300	500–525	65–70	„
„ light „	885–890	..	885–1000	200–250	450–500	75–80	„
„ fluid „	885–890	..	1200–1400	300–350	500–550	40–45	„
„ „ „	885–890	..	900–1000	225–275	450–500	45–50	„

* Standard for viscosity, tallow at 180° F. = 100.

Among the former, or prohibitive, properties we include liability to oxidise or gum, presence of acid and presence of foreign matters or impurities.

Among the latter, or qualificative properties, we include specific gravity, body or viscosity, flashing point, loss by evaporation, setting point and molecular weight, or saponification equivalent, which may, collectively, be regarded as to some extent means of determining the genuineness or sophistication of oils. (See Table D, p. 267.)

Particulars of the processes usually adopted for the determination of the genuineness of oils and fats, for the identification of oils in mixtures, and for the estimation of the fatty acids and foreign matter are given under the heading of "Raw Materials," Chapter V. p. 86. We shall now proceed to refer in order to the properties enumerated above, avoiding repetition as far as possible in the case of those which have been earlier dealt with, but, when necessary, supplementing information already given by details proper to this section, and discussing more fully those properties which have not been previously disposed of. In the course of these remarks we shall have occasion to introduce several tables illustrative of the various properties referred to, with regard to which we take this opportunity of saying that they have been for the most part specially compiled for these pages, and are derived from the examination of a large number of commercial samples, the general characteristics of which afford assurance of their genuineness.

Oxidation or Gumming.—This property depends upon the affinity which different oils exhibit for oxygen, and the readiness with which they absorb it when exposed to the action of the atmosphere. In mineral oils the tendency is almost entirely absent. In animal and vegetable oils it is present in various degrees. A crude method of determining simultaneously the gumming tendency and the body or viscosity of oils is found in placing drops of the oil to be tested at the top of an inclined plane and noting the time occupied and the distance travelled by each before its progress is

TABLE D.

Animal and Vegetable Oils.

Name of Oil.	Sp. Gr. at 60° Fahr.	Viscosity.		Flashing Point. Close Test. Fahr.	Setting Point, about. Fahr.	Free Fatty Acid.	Saponification Equivalent.
		At 70° Fahr.	At 120° Fahr.				
Southern sperm	880·7	100·1	45·4	457·5	41·7	1·32	460
"Bottlenose" or Arctic sperm	880·4	105·3	47·2	446·2	39·2	1·78	463
White whale	920·7	187·7	71·3	476	27·2	2·14	288
Neat's foot..	917	247	82·4	470·3	34·4	7·18	276
Lard	917·2	223·2	79·4	493·9	39·6	2·55	277
Olive	916·7	213·2	75·0	437·5	27	11·15	283
Refined rape, East India seed	916	250·4	88·1	478·6	26·4	2·76	310
" " Black Sea "	920·9	226·9	78·8	465·4	27	5·03	303
" cotton	923·5	190·4	69·8	523	30	0·19	277
Castor	963	2500	390	487	0	1·72	306

Particulars of the samples from which these averages are deduced will be found in the subsequent tables allotted to each property.

arrested by the thickening of the oil; but so many elements of uncertainty are inseparable from this procedure that little confidence can be placed in the results. A more accurate method is to place a weighed quantity of the oil in a shallow porcelain basin, or in a watch-glass, previously tared, which with its contents is to be placed in a water-bath, where it is to be exposed to a temperature of 212° F. for a period of from 12 to 24 hours. Thereafter the vessel and its contents is to be reweighed, and the increase in nett weight will show the amount of oxygen absorbed. It is essential that before submitting oil to this test it should be carefully freed from moisture as the removal of this in the process of this test would falsify the result. Another method is to coat a piece of glass with the oil to be tested, and to keep it for a day at a temperature of 200°–212° F., noting the rate of oxidation as indicated by the increased adhesiveness of the oil when touched with the point of the finger. Similar results may be obtained by exposing films of oil on glass or steel for longer periods, ten to fourteen days, at normal temperatures.

Blown or thickened oils.—The avidity with which certain oils absorb oxygen, and the increase in specific gravity and viscosity by which the process is accompanied, have been taken advantage of to produce artificially thickened oils to be used in imparting body to mineral oils.

The operation may be best effected in a cylindrical pan, the depth of which ought to be from once and a half to twice its diameter, the bottom hemispherical and the top covered. Provision is to be made for heating the contents of the pan, during the earlier stages of the operation, by a steam coil, or by a jacket which need not extend beyond the hemispherical bottom, and it may be convenient to make arrangements whereby, when the steam has been shut off, a stream of cold water may be passed through the jacket or coil in order to check the rise in temperature which takes place after the action of oxidation has fairly begun. From the top of the pan a pipe of liberal dimensions ought to be carried to a flue or chimney, or preferably to a furnace, by

which the acrid fumes which are emitted may be distributed or consumed without causing annoyance to neighbours. The air, which may be propelled by any ordinary force pump, rotary blower, or "centrifugal," * is introduced by a vertical pipe carried nearly to the bottom of the pan and terminating in a coil or cross piece with perforations on the lower side.

The writer introduced another method of oxidising and agitating by air. He dispenses with the coil or cross-piece; the vertical air pipe, open at the bottom, terminates about 1 foot from the bottom of the pan and about 1 inch above the opening in the bottom of an inverted truncated cone, the greatest diameter of which is about one-third of that of the pan, while the opening in the bottom is the same as that of the air pipe. The inverted cone is supported in the centre of the bottom of the pan by stays bolted by short screws to the sides and bottom of the pan. When air is driven through the central pipe it impinges upon the oil at the narrow opening in the cone, and by setting it in motion induces a downward suction and a circulating motion affecting the whole contents of the pan.†

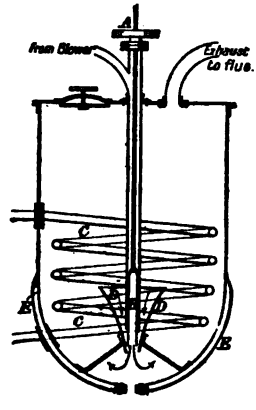


FIG. 80.—J. VEITCH WILSON'S PATENT OXIDISING AND AGITATING PAN.

When oil is to be oxidised the pan is filled to about half its capacity, in order to leave room for ebullition and to prevent the oil from being carried away in the shape of small particles

* Gwynne & Co., Brook Street works, Holborn. E.C., prepare blowing machinery specially for use in the oxidation of oils.

† The writer also used pans of this construction for agitating and blending oils, and for brightening oil which was dull from the presence of moisture.

which are thrown up with the air. Steam is turned into the jacket, or coil, and the temperature of the oil is raised to 160–170° F. When this temperature has been attained the air may be turned on, but, unless arrangements have been made for heating the air to this temperature, it must not be turned on sooner, as cold air would retard the process of heating and prolong the operation of oxidation. At about 170° F. the process of oxidation begins, and by the chemical action which is set up, heat is produced in the oil. When this occurs the steam must be reduced or entirely shut off, and, if the heat of the oil continues to rise too rapidly or too high, the current of air must be reduced or recourse must be had to a stream of cold water through the jacket or coil, as already described.

Authorities differ as to the maximum temperature to be permitted and the time required for oxidation, but as these are affected by the nature and quality of the material operated on, by the volume of air passed through the pan, and by the specific gravity or body required in the product, it is impossible to lay down arbitrary rules in either case.

It is, however, generally understood that the action of oxidation begins at about 170° F., and that the best results in regard to colour and smell of products are obtained by keeping the temperature as low as is consistent with continuance of this action; but as oxidation proceeds more rapidly at higher temperatures, the temperatures adopted in actual working vary from 200° F. to 250° F. according as operators attach more importance to quality of products or to the time occupied in the process, which, subject to the conditions already referred to, may vary from 18 to 48 hours. The oils commonly selected for thickening by oxidation are "Rape Seed" and "Cotton Seed," the latter when thickened being usually known in the trade as "Lardine." The action of oxidation exerts a powerful decolorising influence on these oils, and dispenses with the necessity for chemical bleaching in the case of oils to be subjected to "blowing"; it is also accompanied by a marked increase in the percentage

of free fatty acids and by other changes, as shown in the following table by W. Fox.*

Subject.	Linseed Oil.		Cottonseed Oil.		Rapeseed Oil.	
	Before.	After.	Before.	After.	Before.	After.
Specific gravity ..	·9354	·986	·916	·9685	·927	·985
Free fatty acids ..	2·40	2·73	2·50	3·70	3·20	5·35
Insoluble fatty acids	95·70	87·67	95·43	84·70	95·65	85·50

Various other oils are amenable to this process, e. g. sperm, neatsfoot, lard, olive and ground-nut, but, on account of their higher price and of their lower affinity for oxygen, they do not offer the advantages which are found in oils from cotton seed and rape seed. The sp. gr. of thickened oils ordinarily ranges from 955–960° at 60° F. with a viscosity at 180° F. equal to that of castor oil; to sp. gr. 980–985° at 60° F. with a viscosity at 180° F. equal four to five times that of castor. Blown oils do not readily dissolve in alcohol, but they are completely soluble in petroleum spirit, and their miscibility with mineral oils varies in accordance with the gravity of the blown oil and with that of the mineral oil, the percentage of thickened oil required to form a complete amalgamation rising with the gravities of the oils entering into the mixture. Thus, using a thickened oil of sp. gr. 960° and mineral oil of sp. gr. 885–890° we may find that so low a ratio as 20 thickened oil + 80 mineral oil will blend, while if either or both oils be taken of 20° to 25° higher sp. gr., it is probable that they would refuse to amalgamate in the same proportions, and that some such percentage as 30 thickened oil + 70 mineral oil would be required to form a homogeneous and stable mixture. Cottonseed oil oxidises more easily and more quickly than rapeseed oil, but at equal densities the latter mixes more freely with mineral oil and

* 'Oil and Colourman's Journal,' 1887.

gives a better body for equal percentages, and generally appears to produce a more desirable and satisfactory lubricant. Rosin or rosin oil is occasionally added to blown oils to raise the body and reduce the cost, but such adulterations increase the gumming and drying character of the oil and are to be avoided. Blown oils have been largely used for a number of years, to the extent of 25 to 50 per cent., in the preparation of oils for land and marine engines and heavy machinery, and no serious complaint has been substantiated against them; but it appears to be an anomaly that oils of well-known oxidising tendency like those from cotton seed or rape seed should, after having their gumming and drying tendencies developed by blowing, be adopted as the basis for the production of lubricants, and the writer fears that, unless great care be exercised in removing superfluous oil from bearings on which such oil is used, the slow elimination of the mineral oil by evaporation will produce troublesome deposits on the machinery.

In addition to the direct loss of power and the extra consumption of fuel involved by the drag upon machinery lubricated with oil which tends to gum, the use of such oils involves continual danger from the liability to spontaneous combustion which is present when collections of "waste" or other fibrous material saturated with these oils are permitted to accumulate in the neighbourhood of flues, steam pipes, or even in the ordinary temperatures of mills.

Gellatly obtained the following results by saturating a handful of cotton waste with various oils, wringing well to get rid of superfluous oil, then placing the oily waste in a chamber kept at a temperature of about 170° F., at which, as we have shown, oxidation begins.

Boiled Linseed oil, one sample fired in 75 minutes, another in 105 minutes.

Raw Linseed oil, two samples fired in 4 and 5 hours respectively.

Rape oil. Sample put up at night was found to have been wholly consumed, box and waste, by next morning.

Olive oil. Two samples fired in 5 and 6 hours respectively.

Lard oil fired in 4 hours.

Seal oil fired in 100 minutes.

Sperm oil refused to ignite or to char the waste.

Mineral oils absolutely refused to ignite, and mixtures of 80 "rape" + 20 "mineral" and of 50 seal + 50 mineral did not, when placed in a warm chamber, develop a temperature sufficient to char the cotton.

The other "prohibitive" properties having been dealt with elsewhere, Chap. V., we proceed now to discuss those which affect the value of oils as lubricants.

Specific gravity is commonly, but erroneously, regarded as affording an indication of the class and of the genuineness of an oil, but from Table E, p. 275, in which we show the range of gravity found in different samples of various fatty oils, it will be seen that on one hand there is a variation of several units in the sp. gr. of oils of the same class, while on the other hand the gravities of oils of different classes coincide with, or overlap, one another. Hence mixtures of two or more oils differing essentially in character and value may be made which shall fall within the limits of the sp. gr. of any standard oil, and it follows that, although any considerable departure from the average sp. gr. may be regarded as evidence of adulteration, conformity to standard affords no guarantee of genuineness.

In dealing with the sp. gr. of liquids, distilled water, one imperial gallon of which weighs 10 lb. and occupies, at 60° F. (15·5° C.), 277·27384 cub. in. = 0·16 cub. ft., is taken as standard or unity. In laboratory practice sp. gr. is usually determined by means of small glass flasks or bottles (called "gravity bottles") containing 1000, 500, or 250 grains, or other units, by weight of distilled water at 60° F., and closed by a ground glass stopper perforated by a capillary orifice which permits the escape of any liquid in excess of the exact capacity of the vessel. In determining sp. gr. by this method, the oil to be tested is first brought to

60° F.; a "gravity bottle" is then filled with the oil, the stopper is placed in position, the overflow is carefully wiped off, and the bottle is dried by a soft cloth, care being taken to prevent any variation in the temperature of the contents. The bottle, with its contents, is placed on a chemical balance —a counterpoise being provided for the weight of the bottle —and the weight of its contents is ascertained. When a 1000-grain (water) bottle is used the weight of its contents directly expresses the sp. gr. of the oil in relation to that of water; when a 500 or 250-grain bottle is used the result must be multiplied by two or by four to obtain the relative sp. gr. Thus it is found that the contents of a bottle which, when filled with distilled water at 60° F. weigh 1000 grains, weigh, in the case of castor oil, 963 grains, of colza, 916 grains, and of sperm 881 grains, which are taken as the sp. grs. of these oils, and, water being taken as unity (or 1), the gravities of the oils are expressed as ·963, ·916 and ·881 respectively. The writer, however, having regard to the fact that a gallon of distilled water weighs 10 lb., thinks it more useful to express the gravity of that liquid as 10·00, and of oils in the same manner, as 9·63, 9·16, and 8·81, which gives at once the relative sp. gr. of the oils referred to and the exact weight of one imperial gallon, from which the weight of any number of gallons or the number of gallons in any given weight may be easily calculated.

FIG. 81.—HYDROSTATIC BALANCE.

Another instrument used in laboratories for the determination of sp. gr. is the hydrostatic balance, first introduced by Westphal and shown in Fig. 81. This instrument consists of an "upright" with a cross-head, the right end of

Table E.

Specific Gravities of different samples of various oils at 60° F. = 15·5° C.

Specific Gravity at 60° F.	Southern Sperm.	Arctic Sperm.	White Whale.	Neat's-foot.	Lard.	Olive.	Refined Rape from		Refined Cotton Seed.
							East India Seed.	Black Sea Seed.	
875	..	..	..	..	..	..	..	..	..
876	..	1	..	..	..	..	..	..	..
877	..	..	..	..	..	..	..	..	..
878	7	9	..	..	..	..	..	..	..
879	5	7	..	..	..	..	..	..	..
880	8	28	..	..	..	..	..	..	..
881	12	18	..	..	..	..	..	..	..
882	14	14	..	..	..	..	..	..	..
883	5	3	..	..	..	..	..	..	..
884	1	2	..	..	..	..	..	..	..
885	..	..	..	..	..	..	..	..	..
*	*	*	*	*	*	*	*	*	*
911	..	..	..	..	..	..	..	..	..
912	..	..	..	..	..	..	..	..	..
913	..	..	..	..	..	..	..	..	..
914	..	..	..	1	1	..	11	..	..
915	..	..	..	3	2	3	37	..	..
916	..	..	..	8	5	20	32	..	..
917	..	..	3	9	15	10	24	..	..
918	..	..	5	11	13	8	15	2	..
919	..	..	11	1	3	2	4	6	..
920	..	..	13	2	1	..	..	17	1
921	..	..	12	..	..	..	..	22	1
922	..	..	11	..	..	..	..	10	11
923	..	..	3	..	..	..	..	3	18
924	..	..	1	..	..	..	..	4	29
925	..	..	..	..	..	..	..	..	9
926	..	..	4	..	..	..	..	..	..
927	..	..	1	..	..	..	..	..	..
928	..	..	..	..	..	..	..	..	..
929	..	..	..	..	..	..	..	..	..
930	..	..	..	..	..	..	..	..	..
Total number of samples	52	82	64	35	40	43	123	64	69
Average sp. gr.	880·7	880·4	920·7	917	917·2	916·7	916	920·9	923·5
Allen's—									
Minimum..	875	876	920	914	915	914	914	..	922
Maximum..	884	881	931	916	916	918	916	..	926

which, as shown in the illustration, carries bearings on which a beam is pivoted. From the outer end of this beam a hollow glass cylinder, containing a thermometer, is suspended, while the other end carries a counterpoise exactly equal to the weight of the cylinder and thermometer when both are suspended in air. The arm of the beam carrying the cylinder is divided into ten equal sections marked by incisions on its upper side. The value of a weight placed in any of these incisions is, therefore, in direct ratio to its distance from the centre of the beam. When the cylinder is immersed in water at 60° F., a weight equal to that of the volume of water displaced by the cylinder is suspended from the end of the beam immediately over the cylinder, and equilibrium is maintained. In using this instrument a jar is filled with the liquid to be tested, care being taken that the temperature at the moment of weighing is exactly 60° F. If the liquid under examination be heavier than water, the weight representing water is retained on the balance, and additional weights are placed upon the arm carrying the cylinder and adjusted until the beam is balanced, when the sp. gr. of the liquid is ascertained by adding the value of the extra weights, as indicated by their position on the beam, to the sp. gr. of water represented by 1000. When liquids lighter than water are being tested, the water equivalent is removed from the beam and suitable weights are placed thereon and adjusted in regard to position until the beam is exactly balanced, when the sum of their values indicates the sp. gr. of the liquid.

These methods, however, require more time and delicacy in manipulation than can be afforded in ordinary commercial and manufacturing operations, and for these purposes it is usual to employ instruments called hydrometers, oleometers, or areometers, which being placed in the liquid indicate its sp. gr. by the extent of their immersion as denoted by graduations upon the stem. These instruments, the general form of which is shown in Fig. 82, are usually made of glass, and consist of a graduated stem, a pear-shaped or

cylindrical bulb, and a ball filled with mercury or shot, which acts as a balance-weight. The principle on which they are constructed is that the density and the buoyancy of a liquid are proportionate to its sp. gr., and consequently the instrument will sink in the liquid until the weight of the volume displaced is equal to the weight of the instrument, and is indicated in arbitrary or in relative figures upon its stem. The instruments used in determining the gravities of oils in this way differ mainly in respect to the scale or standard adopted in each, and are briefly described as follows.

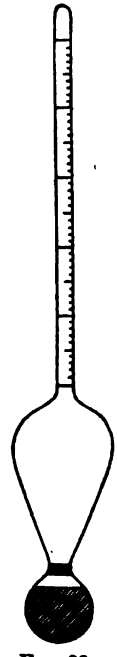


FIG. 82.

The Hydrometer.—This instrument, as its name implies, takes water as standard and expresses the specific gravity of that liquid as 1·000 at 60° F. (15·5° C.). The gravities of liquids heavier than water are represented by higher numerals, and those of liquids lighter than water by lower numerals, but, in both cases, in such a manner as to correspond with the result of determinations by the sp. gr. bottle or by hydrostatic balance, and to indicate simultaneously the sp. gr. of the liquid in relation to that of water and the weight of 1 gallon of the liquid at 60° F., that of 1 gallon of water being 10 lb.

Baumé's Hydrometer or Areometer.—Baumé's object is said to have been to produce an instrument for liquids heavier than water in which each degree should represent a corresponding percentage of salt dissolved in water, and, upon a similar scale, an instrument designed to indicate the density of liquids lighter than water. In the former, which we shall refer to as "H," water is represented by "0," or zero, at the top of the stem; in the latter, which we shall refer to as "L," water is represented by 10 at the bottom of the stem. In each

instrument the ratio is progressive, i.e. in both cases higher numbers are used to register gravity as it recedes, rising or falling, from the standard adopted for water; thus, in "H" the figure 30 equals, say, sp. gr. 1·252, while the figure 30 on "L" equals, say, sp. gr. 879. Much uncertainty obtains with regard to the principle on which Baumé constructed his instruments and graduated their scales, and Prof. Herbert McLeod, author of the article on specific gravity in 'Thorpe's Dictionary of Applied Chemistry,' asserts that there are about two dozen different scales giving equivalents in sp. gr. for "H," and about one dozen giving corresponding equivalents for "L." The greatest divergence occurs in connection with the former, and may be accounted for in various ways.

1st. From differences of opinion as to the standards adopted by Baumé in graduating the instrument.

2nd. From differences in the gravities of standard solutions used in determining fixed points on the instruments.

3rd. From the allotment of uniform spaces upon the stem of the instrument to all the degrees, irrespective of their position on the scale or to alteration in value due to the greater or less immersion of the instrument.

Prof. McLeod believes, and apparently with reason, that, taking water as zero, Baumé fixed the point 15 on "H" by the level at which the instrument floated in a solution of 85 water + 15 common salt at 12·5° C. = 54·5° F., and divided the remainder of the stem into equal intervals, each representing 15 degrees.

Biedermann, in the 'Chemiker Kalender,' holds that Baumé determined the point for 10° on the instrument by noting the level at which it floated in a solution of 90 water + 10 common salt, and divided the remainder of the stem into equal spaces, each representing 10 degrees.

Lunge and Hurter, in the 'Alkali Maker's Pocket-book,' say that concentrated sulphuric acid, sp. gr. 1·8426 at 15° C., gives the point 66 on H, and that the space between that point and zero is to be divided into 66 equal degrees.

The formula for the conversion of degrees Baumé "H"

into sp. gr. is found by multiplying the sp. gr. of the solution taken as standard by the corresponding degrees B., and dividing by sp. gr. − 1 = water, thus taking sp. gr. of 15 per cent. salt solution as 1·1119 (Chandler) = 15° B.; $1{\cdot}1119 \times 15 \div {\cdot}1119 = 149{\cdot}05$ and sp. gr. $= \frac{149{\cdot}05}{149{\cdot}05 - \text{B.}}$.

The incongruities of the system will be seen from the following table, in which we exhibit the actual sp. gr. of salt solutions as determined by Gerlach, and the sp. gr. of corresponding degrees Baumé as given by various authorities.

TABLE showing specific gravity of solutions of common salt in water at 15° C., and specific gravity according to different authorities of corresponding degrees Baumé at 12·5° C., and at 15° C.

Coefficient for expansion between 12·5° C. and 15° C. = ·000246 sp. gr.

Salt Solution.		Degrees Baumé at 12·5° C.	Specific Gravity = Degrees Baumé.			
Parts in 100 of Solution.	S.G. at 15° C., according to Gerlach.		A Chandler at 12·5° C.	B Biedermann at 12·5° C.	C Francœur at 12·5° C.	D Lunge and Hurter at 15° C.
1	1·00725	1	1·00675	1·0069	1·0066	1·007
5	1·03624	5	1·03471	1·0358 ?	1·0340	1·037
10	1·07335	10	1·07191	1·0745 ?	1·0704	1·075
15	1·11146	15	1·11189	1·1160 ?	1·1095	1·116
20	1·15107	20	1·15497	1·1608 ?	1·1515	1·162
25	1·19228	25	1·20153	1·2095 ?	1·1968	1·210

A, Chandler takes 15 per cent. salt solution = sp. gr. 1·11189 = 15 B., giving the formula sp. gr. $= \frac{149{\cdot}05}{149{\cdot}05 - \text{B.}}$.

B, Biedermann proposes to take 10 per cent. salt solution = sp. gr. 1·073596 = 10 B., giving the formula sp. gr. $= \frac{145{\cdot}88}{145{\cdot}88 - \text{B.}}$, but his specific gravities do not agree with this, and appear to agree with those given in column D.

C, Francœur appears to take 15 per cent. salt solution = sp. gr. 1·1095 = 15 B., giving the formula sp. gr. $= \frac{151{\cdot}9}{151{\cdot}9 - \text{B.}}$.

D, Lunge and Hurter take sulphuric acid sp. gr. 1·8426 at 15 C. = 66 B., giving the formula sp. gr. $= \frac{144{\cdot}3}{144{\cdot}3 - \text{B.}}$.

With regard to the graduation of the instrument "L." for liquids lighter than water, with which, in this section, we are more immediately concerned, there is less room for confusion, as it appears to be clearly established that Baumé arrived at his standard for this instrument by taking the point at which it floated in a solution of 90 water + 10 common salt at 12·5° C. as 0, or zero, and the point at which it floated in pure water as 10°, the rest of the stem being divided into equal spaces, each representing 10 degrees. Differences in the conversion of degrees Baumé "L" into sp. gr. can therefore only arise from differences in the gravity taken for the 10 per cent. salt solution, and in the formulæ deduced therefrom for the conversion, and we find that such differences occur.

Gerlach gives the sp. gr. of a 10 per cent. salt solution as 1·07335 at 15° C., in which he is followed by Biedermann. Chandler, in giving the basis of Baumé for light liquids, gives the sp. gr. of a 10 per cent. salt solution as 1·0737665 at 12·5° C. = mean of nine observations, but it is noteworthy that, in his table of equivalents for Baumé for heavy liquids, Chandler gives the sp. gr. 1·07191 as equal 10° B., which ought to indicate the presence of 10 per cent. salt.

The formula for the conversion of degrees Baumé "L" into sp. gr. is arrived at by multiplying the sp. gr., at 12·5° C., of a 10 per cent. salt solution by 10, and dividing by sp. gr. − 1 = water, for numerator, and by dividing sp. gr. of water = 1 × 10 by figures representing the additional sp. gr. due to salt, for denominator. Thus Biedermann gives

$$1{\cdot}073596 \times 10 \div {\cdot}073596 = 145{\cdot}88 \text{ and}$$
$$1{\cdot}00000 \times 10 \div {\cdot}073596 = 135{\cdot}88,$$

giving sp. gr. $= \dfrac{145{\cdot}88}{135{\cdot}88 + \text{B.}}$ The formula derived from Chandler's sp. gr. = 1·07376 is sp. gr. $= \dfrac{145{\cdot}56}{135{\cdot}56 + \text{B.}}$, which practically coincides with Biedermann's, but some American authorities give the formula for conversion of Baumé's "L"

into sp. gr. as sp. gr. = $\frac{140}{130 + B.}$, which points to sp. gr. of 10 per cent. salt solution = zero as 1·0769, which differs considerably from Gerlach's figures. The following table gives Baumé degrees for liquids lighter than water, with equivalents in sp. gr. as given by Chandler, Biedermann, and Brannt.

TABLE showing Baumé's Scale for Liquids Lighter than Water, with corresponding Specific Gravities according to various authorities.

Degrees Baumé.	Specific Gravity.		
	Chandler. Sp. gr. = $\frac{145·56}{135·56 + B.}$ at 12·5° C.	Biedermann. Sp. gr. = $\frac{145·88}{135·88 + B.}$ at 12·5° C.	Brannt. Sp. gr. = $\frac{140}{130 + B.}$ at 15° C.
10	1·00000	1·0000	1·0000
15	·96679	·9669	·9655
20	·93571	·9359	·9333
21	·92974	·9299	·9271
22	·92384	·9241	·9210
23	·91801	·9183	·9150
24	·91226	·9125	·9090
25	·90657	·9068	·9032
26	·90096	·9012	·8974
27	·89542	·8957	·8917
28	·88995	·8902	·8860
29	·88454	·8848	·8805
30	·87919	·8795	·8750
35	·85342	·8538	·8484
40	·82912	·8295	·8235
45	·80616	·8066	·8000
50	·78443	·7849	·7777
55	·76385	·7643	·7567
60	·74432	·7449	·7368
65	·72577	·7262	·7179
70	·70811	·7085	·7000

Correction for temperature: 1° C. = ·00063 sp. gr.; 1° F. = ·00035 sp. gr.; therefore add to Brannt ·00157 to give equivalent of 12·5 C.

The Oleometer.—This old-fashioned instrument, of which there are numerous examples alike unscientific in principle and untrustworthy in use, was commonly at one time, and is even yet to some extent, regarded as a simple means of deter-

mining the genuineness and the value of oils. It is referred to here only to show the fallacy upon which such pretensions are based. In form the instrument corresponds with that of the hydrometers which have been described. The scale is graduated from 0 at the lower end of the stem, which usually corresponds with sp. gr. 970, to 50 at the top, which corresponds with sp. gr. 870, the intervening space being marked off in 100 equal units. The names of half a dozen oils are written or printed upon the paper scale in the stem, and against each a figure is placed, purporting to represent the true gravity of genuine oil, any departure from which is to be regarded as denoting adulteration to a corresponding extent. Unfortunately for the reliance to be placed in these instruments, the writer, who has examined several, of which particulars are given on p. 283, finds that no two agree in the numbers allotted to the oils selected, so that an oil which might be passed as genuine by one, would be condemned as adulterated by another instrument. But, even assuming absolute concordance among all makers of these instruments with regard to the figures to be adopted for each oil, it must still be evident that, on account of the variation which occurs in genuine oils of the same kind, and of the approximation and overlapping which occur in the gravity of oils of different kinds, as shown in Table E, p. 275, no trustworthy conclusion with regard to the value of any oil can be deduced from such an empirical scale.

The gravity of tallow, butter, or any other fatty material which is solid at 60° to 70° F., cannot be conveniently determined by instruments adapted for use at these temperatures, and it is usual in dealing with such articles to have recourse to the "Sprengel tube," which is adapted for use at higher temperatures, and of which a description will be found on p. 97.

Coefficient of expansion.—In the preceding remarks upon sp. gr., reference has frequently been made to 60° F., as although various authorities have preferred to adopt higher or lower temperatures, it is obvious that a uniform standard is essential for general use, and 60° F. = 15·5° C. has

TABLE.

COMPARISON OF OLEOMETERS.

	No. 1.		No. 2.		No. 3.*		No. 4.		No. 5.	
	Scale.	Sp. Gr.	Scale.	Sp. Gr.	Scale.	Sp. Gr.	Scale.	Sp. Gr.	Scale.	Sp. Gr.
Showing actual sp. gr. at 60° F.	50 =	871	50 =	870	50 =	892	50 =	871	50 =	870
= "50" and "0" on each instrument .. :	0 =	970	0 =	975	0 =	1000	0 =	974	0 =	972
Value of unit in sp. gr.	1 =	1·98	1 =	2·1	1 =	2·16	1 =	2·06	1 =	2·04

TABLE

SHOWING THE STANDARD, OR NOMINAL, GRAVITY adopted for various oils in each instrument, and the figure actually indicated by the same instrument when placed in genuine specimens of the oils named.

Sp. Gr. ·60° F.	Name of Oil.	No. 1.		No. 2.		No. 3.*		No. 4.		No. 5.	
		Nominal.	Actual.	Nominal.	Actual.	Nominal.	Actual.	Nominal.	Actual.	Nominal.	Actual.
881	Sperm oil ..	46	45½	46	45	46	55	46	45½	46	45
917	Olive oil	29	25½	27	26½	28	37	27½	27½	28	27½
916	Refined rape oil	28	25½	28	26½	28	37	28	28	28	28
930	Hemp oil ..	..	20½	20	21½	20½	31½	20½	22	20½	21
934	Linseed oil ..	17½	17	17	19	17½	30	17½	20	17½	19
964	Castor oil ..	5	3	..	..	5	17½	5	3½	5	3½

* Several instruments by same maker gave same results.

by common consent been almost universally adopted as the most convenient, except in the case of fats which are not liquid at that temperature. As, however, in some books on the subject, the densities of oils are given at different temperatures, and as in practice, in order to save time or from want of facilities for adjusting temperatures, it may be convenient to take gravities at various temperatures, it is necessary to have some rule for the correction of such departures from standard temperature. For this purpose it was customary in the oil trade to regard 0·0004 (water = 1·000) as the equivalent of 1° F., but Allen, Stillwell and other observers have shown that while the rate of expansion varies in different oils from ·000615 for 1° C. in the case of seal oil to ·000722 for 1° C. in the case of whale oil, the coefficient of expansion in the ordinary fatty oils is fairly uniform and may be safely stated as = ·00063 for 1° C. or ·00035 for 1° F.

Viscosity.—Although the varying thickness or body of different oils cannot have escaped the attention of those who dealt with them practically or experimentally, it is only within recent years that body, or viscosity, has been recognised as a characteristic feature in oils, as a measure of their value as lubricants, and as affording to some extent corroborative evidence in their identification, and that attempts have been made by the introduction of suitable instruments, and by the adoption of common standards, to reduce this property to a satisfactory basis. Various apparatus, too numerous to mention in detail, have been designed for the purpose of determining this property. In the majority of these instruments the principle adopted is a modification of the sand glass, i. e. the passage of a fixed quantity of oil through a narrow orifice. Among other principles we find the syphon (adopted by Professor Osborne Reynolds); capillarity, by noting the rate of flow through strands of yarn of equal size and number; the use of a paddle as a measure of resistance, and, on the same principle, the revolution in the oil of a series of vertical concentric cylinders operated by a falling weight (James Napier).

Among the instruments of the more numerous class, viz. those constructed on the principle of running or dropping a given quantity of oil through a narrow orifice, only two have obtained any general acceptance. The older instrument consisted originally of a graduated glass burette, carried on a stand in a vertical position, the time taken by various oils to fall from a higher to a lower mark being taken as their viscosity. By and by, in order to permit of the regulation of the temperature, the burette was enclosed in a wider glass cylinder or jacket which was filled with water, for heating which various arrangements were adopted.

This instrument presented several objectionable features. Being made of glass, it was liable to be broken by accidents in handling or by sudden variations in temperature. It was difficult or impossible to produce different instruments of uniform standard, and it was found that on account of the length of the tube and the height of the column of oil, the sp. gr. of the latter exercised an irregular influence upon the flow of different oils and upon their apparent viscosity. To obviate these defects Boverton Redwood adopted an instrument which had been designed by the late W. H. Hatcher, formerly manager of the Belmont Works of Price's Patent Candle Company, and which had for some time been in use at those works.

This apparatus consisted of an inner and an outer copper cylinder of 1¾ in. and 5½ in. diameter respectively, and about 3 in. deep. These, being attached to a common bottom, formed an inner chamber for the oil to be tested, and an outer chamber or bath which was filled with water at any required temperature. In the centre of the bottom of the inner chamber an agate nipple was inserted, through which a small orifice was drilled to permit the efflux of the oil.

The advantages offered by this instrument were that the risk of breakage was eliminated, that the greater area of the oil chamber permitted a reduction in the depth or "head" of oil with a corresponding reduction in the apparent influence of sp. gr. upon viscosity, and that the use of an

agate nipple rendered it possible by drilling to produce orifices of more uniform size than can be obtained in glass tubes.

The instrument was, however, still far from fulfilling the conditions required in one which could be regarded as a standard, and to overcome the defects which were present, Redwood devised and introduced the following important improvements:—

1. A projecting tube attached to the outer cylinder by which the contents of the bath may be heated and maintained at any required temperature.

2. A paddle by which the contents of the bath may be gently agitated and the temperature equalised.

3. A curved shield which partly covers the bath and prevents the liquid in the latter from splashing into the oil cylinder.

4. A projecting upturned point or gauge by which the height of oil in the inner cylinder is simply and accurately regulated, and the standardising of the instrument is facilitated.

The Redwood viscosimeter, which has been almost universally adopted as a trustworthy and convenient means of determining and expressing the body or viscosity of oils, is shown in A, B, Fig. 83. It consists of an outer and inner cylinder, as has been described, the space between these forms a bath, the contents of which are heated by the application of a flame to the tube projecting on the right side, and are drawn off, when necessary, by the cock shown on the left side. The bath is provided with a paddle consisting of four helical blades attached to a loose sleeve enclosing the inner cylinder, and operated by a small handle attached to the upper curved portion of the sleeve. In this upper portion a small tube is fixed through which a thermometer is inserted to indicate the temperature of the bath. The inner cylinder is the oil container; about the middle a rectangular wire is fixed, terminating in a sharp upturned point which indicates the level of the surface of the oil at the beginning of a test. The outflow of the oil is through a narrow orifice,

about ·072 in. diameter, drilled in an agate nipple fixed in the bottom of the chamber. The upper side of the agate forms a cup, and the orifice is closed by a ball attached to a

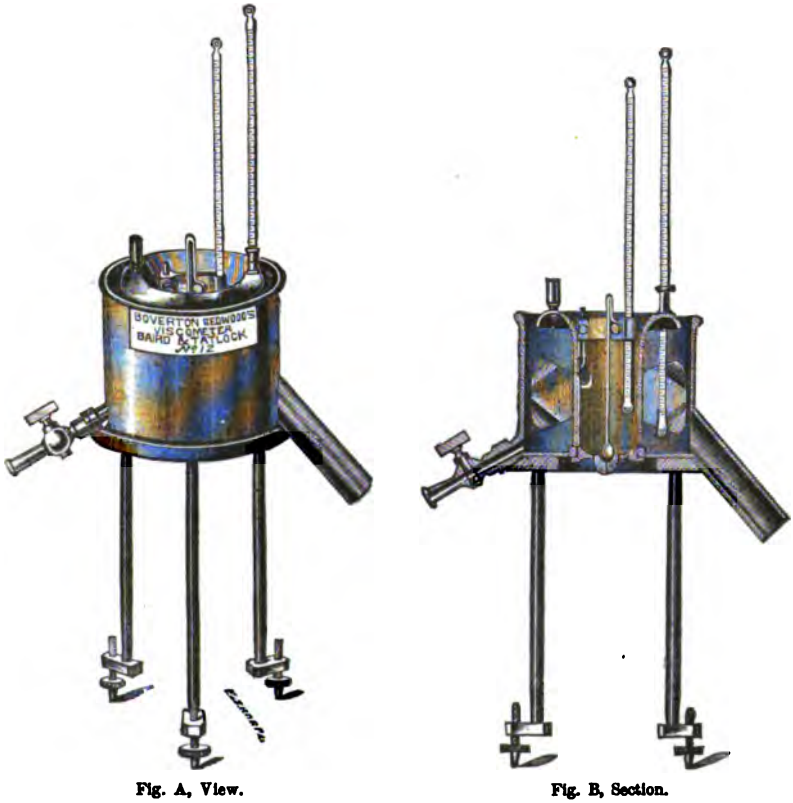


Fig. A, View. Fig. B, Section.

FIGS. 83.—REDWOOD'S VISCOSIMETER.

wire, by which it is raised when the experiment is to be started. To the upper side of the oil cylinder an arm is fixed, projecting inwards, which serves to carry a thermometer indicating the temperature of the oil, and upon

which the ball valve is suspended when a test is in progress. The oil is received in a narrow-necked flask containing 50 *c.c.* when filled to a mark on the neck, the flask being placed in a beaker or other vessel containing water, which is maintained at the same temperature as that of the oil and water in the vessels above.

In using the instrument the water bath is brought to the temperature required (for temperatures above 200° F. a heavy mineral oil may be substituted for water), the oil to be tested is brought to the same temperature and is transferred to the oil chamber, the height being determined by the wire gauge, where it is kept till the thermometer in the oil indicates the same temperature as that in the bath. The orifice in the bottom of the oil chamber is opened, a stop-watch or chronograph is simultaneously started, and the time required for 50 *c.c.* to flow into the receiver below is noted. Each oil ought to be tested twice at least, and oftener if the first results are discordant.

For the purpose of registration it is customary to compare the results with the viscosity of some standard oil such as refined rape or preferably sperm oil * which exhibits greater constancy in respect to viscosity than rape oil, and for simplicity in reference the actual figures obtained may be multiplied by 100 and divided by the corresponding figures for the oil adopted as standard, which is then to be expressed as 100, the viscosity of the oil under examination being expressed in multiples or in fractions thereof as is done in Table F., on p. 289.

Redwood recommends that a correction be made for the effect of sp. gr. by multiplying the viscosity of the oil by its sp. gr. and dividing the result by the sp. gr. of the standard oil. Thus the viscosity of refined rape oil being 246 and its sp. gr. 916, while sperm oil has viscosity = 100 and sp. gr. 881, $246 \times 916 = 225{,}336 \div 881 = 256$, the latter figure being taken as the corrected viscosity of refined rape oil.

* Sperm oil is adopted as standard for viscosity in this chapter, being taken as = 100 at 70° F.

Table F.

Specific Viscosities of different samples of various oils at 70° F. = 21° C. Standard, Southern Sperm = 100 vis.

Viscosity at 70° F.	Southern Sperm.	Arctic Sperm.	White Whale.	Neat's-foot.	Lard.	Olive.	Refined Rape from		Refined Cotton Seed.
							East Indian Seed.	Black Sea Seed.	
90	..	2	..	..	..	..	..	..	..
95	12	2	..	..	..	..	..	..	..
100	27	10	..	..	..	..	..	..	..
105	11	49	..	..	..	..	..	..	..
110	1	17	..	..	..	..	..	..	..
115	..	4	..	..	..	..	..	..	..
120	..	..	..	..	..	..	..	..	..
*	*	*	*	*	*	*	*	*	*
160	..	..	1	..	..	..	..	..	..
165	..	..	..	..	..	..	..	..	..
170	..	..	4	..	..	..	..	..	..
175	..	..	4	..	..	..	..	..	..
180	..	..	8	..	..	..	..	..	5
185	..	..	11	..	..	..	..	..	24
190	..	..	11	..	..	..	..	..	16
195	..	..	13	..	1	..	..	..	11
200	..	..	10	..	..	1	..	..	10
205	..	..	..	..	1	4	..	4	3
210	..	..	..	..	2	15	..	3	..
215	..	..	..	..	3	7	..	7	..
220	..	..	..	1	12	8	1	6	..
225	..	..	..	..	10	1	4	13	..
230	..	..	..	2	3	1	1	8	..
235	..	..	..	4	2	..	4	12	..
240	..	..	..	2	1	..	20	9	..
245	..	..	..	2	..	..	15	1	..
250	..	..	..	6	1	..	23	..	..
255	..	..	..	4	1	..	19	1	..
260	..	..	..	2	..	..	22	..	..
265	..	..	..	..	..	..	12	..	..
270	..	..	..	1	..	..	1	..	..
275	..	..	..	1	..	..	1	..	..
280	..	..	..	..	..	..	..	..	..
Total number of samples..	51	84	62	25	37	37	123	64	69
Average viscosity.. ..	100·1	105·3	187·7	247	223·2	213·2	250·4	226·9	190·4

But the writer considers that for practical purposes it is unnecessary and delusive to attempt to differentiate between the viscosity due to the cohesive properties of the oil and that which may be due to its heavier or lighter sp. gr., and in his own practice and in the tables of viscosity given in this chapter, he adopts the figures obtained by direct determination as the true viscosity of an oil rather than figures denoting what its viscosity might be if its gravity coincided with that of some other oil.

Flashing Point.—In the earlier days of the mineral oil trade, no official recognition was given to the flashing point of these oils or to the methods adopted in determining it, and, as the instruments employed for the purpose and the methods of using them differed considerably, the results were irregular and unsatisfactory.

In 1871 the first British "Petroleum Act" fixed the minimum flashing point for safety at 100° F., and described an instrument by which it should be determined, but after a few years' experience it was found that, from defects in the instrument itself and from the absence of definite regulations for its use, it failed, especially in the hands of different and inexperienced operators, to give concordant results.

In 1879 the second Petroleum Act was passed, by which a new apparatus designed by Sir Frederic Abel was constituted, and still continues the official standard for the United Kingdom for testing mineral burning oils. By the same Act the minimum flash point, which by the former Act had been fixed at 100° F., "open test," was reduced to 73° F., as being, by the new "close test" apparatus, the equivalent of the old standard. Neither of these instruments, in which a water bath is an essential part, are suitable for testing lubricating oils, the flashing points of which ought to be, and usually are, much above the boiling point of water. Nor has Government in any way attempted to regulate the flashing points of these oils which far exceed the minimum required for public safety. But as the inflammability of lubricating oils is a matter of importance to those who use them, to insurance companies,

Table G.

Flashing Points of different samples of various oils by "Close" Test.

Flashing Point. Fahr. "Close" Test.	Southern Sperm.	Arctic Sperm.	White Whale.	Neat's-foot.	Lard.	Olive.	Refined Rape from		Refined Cotton Seed.
							East Indian Seed.	Black Sea Seed.	
380	..	..	..	..	..	..	..	..	..
385	..	..	..	..	..	..	..	..	..
390	..	2	..	..	..	..	..	..	..
395	..	..	..	..	..	..	..	..	..
400	..	1	..	..	..	..	..	..	..
405	..	..	..	..	..	..	..	..	..
410	..	1	..	2	..	1	2	..	..
415	..	1	..	2	..	1	..	..	..
420	1	4	..	..	..	3	..	..	..
425	..	3	..	1	1	1	..	..	..
430	..	3	1	..	..	4	2	1	..
435	2	..	..	1	1	4	..	..	..
440	2	7	3	1	..	1	2	2	..
445	4	7	3	..	..	1	..	..	..
450	2	5	1	..	1	4	1	2	..
455	6	9	1	..	..	..	2	2	..
460	6	2	3	..	..	3	5	4	..
465	3	4	2	1	..	1	3	2	..
470	3	4	3	2	..	..	7	2	..
475	..	2	2	..	..	..	8	1	..
480	3	3	3	..	..	..	18	8	..
485	2	1	..	1	3	..	5	..	..
490	..	..	2	..	1	..	18	1	..
495	..	..	2	..	1	..	7	..	..
500	..	..	3	..	1	..	5	..	2
505	..	..	..	..	3	..	..	..	2
510	..	..	3	1	1	..	4	..	1
515	..	..	2	..	2	..	..	..	2
520	..	..	..	2	2	..	..	..	2
525	..	..	..	..	..	..	..	..	3
530	..	..	1	1	..	..	..	..	5
535	..	..	..	1	..	..	..	..	2
540	..	..	..	1	..	..	..	..	3
545	..	..	..	..	1	..	..	..	..
550	..	..	..	..	..	..	..	..	..
Total number of samples	34	59	35	17	18	24	89	25	22
Average flash point ..	457·5	446·2	476	470·3	493·9	437·5	478·6	465·4	523

and to public carriers, this property is not less carefully observed by those who are interested in such oils, although it escapes official recognition.

Various modifications of the apparatus used in testing burning oils have been introduced in order to adapt them

FIG. 84.—GRAY'S FLASH POINT APPARATUS.

for testing lubricating oils. Among these the most u[illegible] are the Pensky-Martin apparatus manufactured in B[illegible] and Gray's, which is made in this country.* The lat[illegible] shown in Fig. 84, A and B. This instrument co[illegible]

* By Baird & Tatlock, 14 Cross Street, Hatton Garden, London,

of a brass cup of same size as that used in the Government standard petroleum tester, viz. 2 in. diameter, by $2\frac{2}{10}$ in. in depth. A line cut round the inside of the cup, $1\frac{1}{2}$ in. from the bottom, indicates the height to which it is to be filled in testing oil. The cup is closed by a tightly fitting cover, through the centre of which a small steel shaft or spindle passes to the bottom of the cup, carrying two sets of stirrers, one of which is below the surface of the oil, the other in the vapour space above. On the top of the spindle, above the lid, a small bevelled wheel with milled edge is fixed, which gears with a vertical bevelled wheel on the inner end of a horizontal shaft carried on two standards fixed on the lid, and terminating at the outer end in a disc of non-conducting material in which is fixed a handle for rotating the shaft. There are four openings in the lid—through one a thermometer is inserted to indicate the temperature of the oil, the remaining three provide means of producing the flash; one of these is immediately in front of the small tilting lamp which ignites the gases, the other two, one on each side, admit air to produce the explosive mixture. These ports are normally closed by a loose, three-quarter disc, flat cover, provided with openings which, when the cover is turned one quarter round, coincide with the ports in the fixed lid.

In using this instrument, the cup is filled to the mark with the oil to be tested, the test lamp is lighted, the flame being adjusted to about $\frac{1}{8}$ inch in size. Heat is applied below the oil cup by a gas or spirit flame, either direct or through the medium of a sand bath, and the temperature of the oil is raised at the rate of 10° to 15° F. per minute, the stirrers being turned at short intervals during the process of heating. When nearing the point at which the oil is expected to flash, it is advisable to reduce the rate of heating, in order that observations may be more accurately made.

The test for flashing point is made by drawing the horizontal shaft, which has about $\frac{1}{8}$ inch end play, outwards, which puts it out of gear with the stirrers and in gear with the sliding cover, which, being partly turned, opens the

ports and tilts the flame of the test lamp into the centre one. If gas is present in sufficient quantity, a slight flash and explosion are produced, if not, the ports are automatically closed, the heating is continued, the stirrers being used, and the test for "flash" repeated at intervals till the flash is observed, the temperature at which this occurs being taken as the flashing point of the oil. For flashing points of standard oils, see Tables A, B, C, D, pp. 263, 265 and 267, and Table G, p. 291.

Evaporation.—This property is closely associated with that of flashing point, which has just been discussed, inasmuch as both depend upon the readiness with which oils may be vaporised at certain temperatures. Two methods have been adopted for the determination of the property of evaporation. The advocates of one system profess to be able to determine the temperature at which evaporation begins, and refer to this as the "evaporating point" of the oil. The advocates of the other system prefer to note the loss sustained by various oils under conditions which are to be regulated in accordance with the class of oil under consideration, or by the purpose for which the oil is intended. As, however, animal and vegetable oils seldom lose weight unless exposed to a temperature approaching that at which decomposition commences, and at lower temperatures are more likely to increase in weight by oxidisation, it is obvious that the determination of the property of evaporation is important only in connection with mineral oils. But from the complex nature and the various boiling points of the different fractions which are found in apparently homogeneous oil, it is evident that, in the case of mineral oils, evaporation may begin, and may continue, at temperatures much lower than that at which the density of the liberated vapours render them visible to the most acute perception, or sufficient to affect the most delicate instruments.

It appears, therefore, to be more in accordance with scientific accuracy and of greater practical utility to determine the amount of loss by evaporation under regulated

conditions of time and temperature, rather than the temperature at which the action of evaporation first becomes visible. The method ordinarily adopted for determining loss by evaporation corresponds with that described under the head of oxidation: a weighed quantity of oil is placed in a tared porcelain basin or in a watch glass, the vessel with its contents is placed in a water or air bath, and exposed for a few hours to a fixed temperature, after which the vessel is reweighed, the tare subtracted, the difference in nett weight indicating the loss (or gain) during the test.

In the case of oils intended for the lubrication of machinery at normal temperatures, exposure for five hours to a temperature of 210°–212° F., which may be obtained in an ordinary water bath, ought to be sufficient, but in the case of cylinder oils, or of oils intended for use on bearings of machinery working at steam temperatures, the tests ought to be made at a temperature corresponding with a steam pressure of not less than 150 lbs., say 360° F.

L. Archbutt has introduced an improvement on this process, by which he also supersedes the ordinary method of determining flash points. Through an oven heated by gas jets he carries a metal tube of $\frac{7}{8}$ in. internal diameter. In this tube he introduces another of glass, the external diameter of which just permits it to pass through the metal tube. In the middle of the glass tube he places a platinum boat, tared, and containing 0·5 *grm.* of oil. In the upper part of the oven there is a coil of pipes through which he passes a current of air, at the rate of 2 *litres* per minute, which attains the same temperature as the oven, and is then by a suitable connection made to pass through the tube in which the vessel containing the oil has been placed. After one hour's exposure the boat containing the oil is withdrawn, reweighed, and the loss noted. The method is said to give accurate and trustworthy results.

Setting Point.—The importance of having an oil which may be exposed to a moderate degree of cold without solidifying, arises partly from the inconvenience of handling an

Table H.

Setting Points of different samples of various oils.

Setting Point. Fahr.	Southern Sperm.	Arctic Sperm.	White Whale.	Neat's-foot.	Lard.	Olive.	Refined Rape from		Refined Cotton Seed.
							East Indian Seed.	Black Sea Seed.	
Ceasing to flow in ½-in. tube when laid on its side at 15	..	..	2	..	..	..	..	..	..
16	..	..	..	..	..	..	1	1	..
17	..	..	..	..	..	..	..	..	..
18	..	..	..	..	..	..	2	..	..
19	..	..	..	..	..	..	..	..	..
20	..	..	4	..	..	2	4	1	..
21	..	..	1	..	..	2	2	..	..
22	..	..	..	..	..	..	1	..	..
23	..	..	..	..	..	..	1	..	..
24	..	..	..	..	..	1	2	..	..
25	..	..	..	..	..	1	8	1	1
26	..	..	..	..	..	..	6	4	..
27	..	..	..	..	..	3	..	1	1
28	..	..	..	..	..	3	3	2	1
29	..	1	2	..	..	2	..	..	..
30	..	..	1	2	..	1	24	7	2
31	..	..	1	..	..	..	..	..	..
32	..	..	..	2	..	..	..	..	2
33	..	..	3	..	..	..	..	..	1
34	..	..	..	..	..	..	..	..	1
35	..	2	1	1	1	..	..	..	..
36	1	2	..	..	1	1	..	..	..
37	..	..	..	..	..	..	..	..	..
38	2	4	1	..	..	..	..	..	..
39	1	5	..	..	1	..	..	..	..
40	2	3	1	..	1	1	..	..	..
41	..	1	..	2	..	..	..	..	..
42	1	2	..	..	..	..	..	..	..
43	1	2	..	..	..	..	..	..	..
44	..	..	..	..	..	..	..	..	..
45	4	1	..	..	..	..	..	..	..
46	1	..	..	..	..	..	..	..	..
47	..	..	..	..	..	..	..	..	..
48	..	1	..	..	1	..	..	..	..
49	..	..	..	..	..	..	..	..	..
50	..	..	..	..	..	..	..	..	..
Total number of samples	13	24	17	7	5	17	54	17	9
Average settling point	41·7	39·2	27·2	34·4	39·6	27	26·4	27	30

oil which is liable to "set" in a cask or tank at the temperatures to which it may be exposed in an ordinary store-room or warehouse, and partly from the danger of damage to machinery if the oil should become solid in the lubricators, through which it ought to be applied to the bearings.

The setting point is probably one of the least constant properties of oils, depending as it does upon the materials from which they are obtained, upon the season of the year in which they are produced, and on the temperature to which they have been subjected in the process of manufacture, by which the percentage of stearin present in the case of animal or vegetable oils, and of paraffin in the case of mineral oils, is regulated. Further irregularities are introduced by the different interpretations applied to the expression "setting point," some regarding it as the temperature at which oils become turbid and cease to flow in a bottle when laid on its side, others, the temperature at which the oil becomes so consistent that it will withstand gentle pressure on the surface or slight concussion on the side of the bottle without giving way.

L. Archbutt uses a leaden cylinder 6 in. diameter by 14 in. deep, placed in a wooden box 12 in. square by 20 in. deep, the space around and below the cylinder being filled with cotton waste as a non-conductor. In the bottom of the cylinder he places a freezing mixture, a number of ½-in. test tubes are filled with the oils to be tested, and corked; another test tube is filled with an oil of known low freezing point, and fitted with a perforated cork through which a thermometer is inserted. The tubes are simultaneously placed in the freezing mixture, from which they are at intervals taken out for examination and returned, till the setting point of each has been reached, the point being that at which after the oil has become apparently solid it refuses to yield when a gentle tap is administered to the side of the tube.

Free Fatty Acids.—The nature of, and the means of determining, free fatty acids in oils have been dealt with in the chapter upon Raw Materials, No. IV., p. 89. We refer

Table J.

Free Fatty Acids in different samples of various oils.

Free Fatty Acid, not exceeding	Southern Sperm.	Arctic Sperm.	White Whale.	Neat's-foot.	Lard.	Olive.	Refined Rape from		Refined Cotton Seed.
							East Indian Seed.	Black Sea Seed.	
per cent.									
0·1	..	..	..	..	..	..	..	..	8
·25	..	..	..	1	..	..	..	..	7
·50	1	3	..	..	..	..	..	..	1
·75	2	1	..	..	..	..	1	..	..
1	4	..	4	..	1	..	..	..	..
1·5	3	8	6	3	4	..	3	1	..
2	4	11	4	2	5	..	13	1	..
2·5	..	5	2	1	..	..	25	..	..
3	..	1	2	..	..	..	22	..	..
3·5	..	..	1	..	..	..	8	1	..
4	..	..	1	..	..	2	4	5	..
4·5	..	..	..	..	..	..	2	3	..
5	..	..	..	..	..	1	1	8	..
5·5	..	..	1	..	..	1	..	4	..
6	..	..	..	..	..	1	..	4	..
6·5	..	..	..	..	..	..	..	..	..
7	..	..	..	1	..	1	..	..	..
7·5	..	..	..	1	..	..	..	1	..
8	..	..	..	..	..	1	..	1	..
8·5	..	..	..	..	..	..	..	..	..
9	..	..	..	1	..	2	..	1	..
9·5	..	..	..	..	..	..	..	..	..
10	..	..	..	1	..	3	..	..	..
11	..	..	..	1	1	7	..	..	..
12	..	..	..	..	..	1	..	..	..
13	..	..	..	1	..	5	..	..	..
14	..	..	..	..	..	2	..	..	..
15	..	..	..	1	..	..	..	..	..
16	..	..	..	..	..	1	..	..	..
17	..	..	..	..	..	1	..	..	..
18	..	..	..	..	..	..	..	..	..
19	..	..	..	..	..	..	..	..	..
20	..	..	..	..	..	1	..	..	..
21	..	..	..	..	..	..	..	..	..
22	..	..	..	..	..	..	..	..	..
23	..	..	..	..	..	1	..	..	..
24	..	..	..	1	..	..	..	..	..
25	..	..	..	..	..	..	..	..	..
Total number of samples	14	29	21	15	11	31	79	30	16
Average F.F.A. per cent. ..	1·32	1·78	2·14	7·18	2·55	11·15	2·76	5·03	0·19

to the subject now, mainly to draw attention to the accompanying Table J, in which the maximum, minimum, and average percentage of free fatty acids in various oils are shown.

The injury which must happen to machinery from the use of lubricants containing any appreciable quantity of mineral acid, is so generally recognised as to render comment here unnecessary. It is, however, less commonly understood that, although in animal or vegetable oils which have been refined with ordinary care, mineral acids are seldom present, the majority of such oils contain free fatty acids in sufficient quantity to act injuriously upon metal, for evidence of which we refer to the table on p. 298.

It is true that by treatment with a weak alkaline solution the free fatty acids may be converted into soapy magma and removed from the oil, but as the loss of oil by this process considerably exceeds the quantity of free fatty acid present, and as the magma is of comparatively little value, the process is adopted to only a limited extent, the operations of oil refiners being mainly directed to improvement in the colour and smell of, and to the removal of foots or other mechanical impurities from the oils in which they deal.

Saponification Equivalent or Molecular Weight.—The fact that all animal and vegetable oils contain the elements of fatty acids and glycerin, and that the fatty acids of different oils are distinguished by different combining weights, i. e. by the number of units of each required to neutralise an equivalent of an alkali (e. g. 56 units of caustic potash KHO; 40 units of caustic soda NaHO, &c.), has been adopted by Koetstorfer as the basis of a method of obtaining corroborative evidence of the genuineness of an oil. Koetstorfer's process is fully described under "Identification of Oils in Mixtures" on p. 106.

By the following modification, by which all the fatty matter is converted into fatty acids, the danger of error due to irregularity in the free fatty acid in the original oil is eliminated, and it is claimed that more accurate results may be

Table K.

Saponification Equivalents of fatty acids of different samples of various oils.

Saponification Equivalent.	Southern Sperm.	Arctic Sperm.	White Whale.	Neat's-foot.	Lard.	Olive.	Refined Rape from		Refined Cotton Seed.
							East Indian Seed.	Black Sea Seed.	
260	..	..	..	..	..	..	..	..	..
265	..	..	..	..	..	..	..	..	..
270	..	..	..	3	..	..	..	..	1
275	..	..	1	15	12	6	..	..	9
280	..	..	..	6	6	7	..	..	4
285	..	..	7	..	..	4	..	..	1
290	..	..	13	..	..	5	..	..	..
295	..	..	3	..	..	..	..	..	..
300	..	..	..	..	..	1	4	4	..
305	..	..	..	..	..	..	6	3	..
310	..	..	..	..	..	..	15	1	..
315	..	..	..	..	..	..	8	..	..
320	..	..	..	..	..	..	2	..	..
325	..	..	..	..	..	..	..	..	..
330	..	..	..	..	..	..	1	..	..
335	..	..	..	..	..	..	..	..	..
340	..	..	..	..	..	..	..	..	..
345	..	..	..	..	..	..	..	..	..
350	..	..	..	..	..	..	..	..	..
430	..	2	..	..	..	..	..	..	..
435	..	1	..	..	..	..	..	..	..
440	1	1	..	..	..	..	..	..	..
445	2	1	..	..	..	..	..	..	..
450	2	1	..	..	..	..	..	..	..
455	3	2	..	..	..	..	..	..	..
460	8	8	..	..	..	..	..	..	..
465	5	11	..	..	..	..	..	..	..
470	4	10	..	..	..	..	..	..	..
475	1	2	..	..	..	..	..	..	..
480	..	5	..	..	..	..	..	..	..
485	..	..	..	..	..	..	..	..	..
490	..	..	..	..	..	..	..	..	..
495	..	..	..	..	..	..	..	..	..
500	..	..	..	..	..	..	..	..	..
Total number of samples	26	44	24	24	18	23	36	8	15
Average sap. equiv. ..	460	463	288	276	277	283	310	303	277
Allen	380–454	419–456	250–303	299	285–296	285–296	314–328	..	285–294

obtained. About 20 *grm.* of the fat or oil are saponified by heating in a basin with an excess of a strong solution of alcoholic potash. The solution is evaporated nearly to dryness, the soap is dissolved in water and decomposed by the addition of an excess of mineral acid. The solution is boiled gently until the fatty acids appear as a transparent layer on the surface of the solution, the latter is removed by syphon or otherwise, the fatty acids washed repeatedly with water and dried. A known weight is then dissolved in alcohol and the solution titrated, in the presence of phenol phthalein as an indicator, with an exactly standardised solution of alcoholic potash.

If 10 *grm.* of the acids require 36·30 *c.c's.* of normal caustic potash solution (1 *c.c.* = 56 *mg.* KHO), then we have for the saponification equivalent 10 *grm.* or 10,000 *mg.* ÷ 36·30 = 275·5, i.e. 56 *mg.* of KHO neutralise, or are equivalent to, 275·5 *mg.* of fatty acid.

The saponification equivalents given in Tables D, p. 267, and K, p. 300, have been determined by the latter process, but it is necessary to point out that as the molecular weights of oils of the same class vary, while those of oils of different classes approach and overlap one another, the test cannot by itself be regarded as final evidence of the genuineness of any oil or of the adulteration of one fatty oil with another, but as no saponifiable matter is present in hydrocarbon oils, the process may be usefully employed in detecting the presence, and in determining the percentage, of mineral oil or of rosin oil in supposed fatty oils or in mixtures.

Having discussed the properties which affect the value of oils as lubricants, it only remains for us to indicate briefly the practical importance attaching to each, and the principles upon which lubricants ought to be selected for different kinds of machinery and for different conditions.

We have already referred to the properties of oil as "negative or prohibitive" and as "positive or "qualificative," and, following this division, we have now to say that any oils which exhibit in a pronounced degree the properties embraced under the former heading, viz.: tendency to oxidise

or gum, presence of acids or of mechanical impurities, are at once to be rejected.

Regarding the properties which we distinguish as "qualificative," we submit the following remarks:—

Specific Gravity has little or no practical value in relation to the lubricating power of an oil, but may serve, in the case of any considerable departure from standard, to direct attention to adulteration.

Flashing Point is a measure of safety. In the earlier days of the mineral oil trade there was difficulty in producing oils of good colour with high flash point, but the necessity for removing the lighter and more volatile fractions, and the greater care with which this is now accomplished, produce, in the majority of cases, oils the flashing points of which amply conform to any standard imposed by practical conditions. Mineral lubricating oils which will not evolve inflammable vapour under temperatures ranging from 350° to 400° Fahr. (open test) may be easily obtained, and, as even the lower temperature can never be attained generally, except in the case of actual conflagration, and is not likely to be attained locally, even in the case of heated bearings, without attention being called to it and without measures being taken to reduce the temperature, little or no danger can be apprehended from the use, under ordinary circumstances, of oil having a flashing point of 350° F. (or upwards), and this figure is commonly accepted by insurance companies and by oil users and manufacturers as a reasonable minimum for mutual protection.

In the case of cylinder oils the importance of high flashing point as a measure of safety is less obvious, since inflammable vapours, if any, must be liberated in the presence of steam, which excludes air and prevents the possibility of either ignition or explosion. As a measure or index of evaporation, the flashing point of cylinder oils is a matter of great importance, and is referred to in the following paragraph. It may, however, be explained that in cylinder oils, as in other mineral oils, high body or viscosity usually involves high

flashing point, as, in order to obtain the required body, the better grades of cylinder oil are subjected to the action of close and open superheated steam, by which they are maintained for hours at a temperature of about 600° F., until all the fractions boiling at that temperature have been removed and only a trace of anything volatile under that temperature remains. A minimum flash point of 500° F., open test, may therefore be reasonably required in any cylinder oil, and good oils will offer 25° to 50° higher.

Evaporation.—It is evident that an oil which when exposed to normal working conditions loses any appreciable percentage of its bulk by evaporation, is to that extent unprofitable, and is probably an unsatisfactory lubricant. This condition applies mainly to cylinder oils which, especially in modern engines, are frequently exposed to temperatures approaching the boiling point of the lighter hydrocarbons, and it is obvious that unless these have been thoroughly removed in the preparation of the oil, loss must ensue from its use. A number of samples of oils used in cylinders were exposed in shallow porcelain basins to a temperature of 360° F. = 150 lb. steam pressure, for a period of five hours, and gave the following results:—

6 filtered oils lost, 0·02, 0·03, 0·07, 0·12, 0·41, 0·69 per cent.

5 natural oils lost, nil, nil, 1·99, 5·73, 6·73 per cent.

2 natural oils gained, 0·11, 0·37 per cent.

L. Archbutt, who recognises the superior importance of the property of evaporation as compared with flashing point in the case of cylinder oils, has superseded the ordinary test for flash point by the test for evaporation described on p. 295, and regards 1 per cent. loss as the maximum permissible after exposure for one hour to a temperature of 370° F.

Setting Point more immediately affects the convenience of those who are required to handle and distribute oil, though it may also in some cases involve trouble and damage. The inconvenience likely to arise from the use of oils which are

liable to "set" and to refuse to flow from a cask or tank at normal temperatures is apparent, and engineers do not require to be reminded of the damage which may accrue to bearings before sufficient heat has been generated to melt oil which has frozen in the lubricators, or to cylinders or valves if the lubricant be too thick to find its way through the narrow orifices and tubes by which it is conveyed to the cylinder or steam chest.

Composition.—Great differences of opinion exist, and will continue to exist, regarding the most suitable materials for use as lubricants, and, on account of the numerous subordinate and collateral considerations requiring attention, e. g. the effect of the lubricants upon materials with which they may accidentally come into contact, it is impossible to lay down any arbitrary rules as to the use of oils or their allotment to particular classes of work. It is, perhaps, of more practical importance to say that from an examination of many hundreds of samples representing oils used for lubrication in the chief manufacturing districts of the United Kingdom, the following facts were deduced :—

1. That in an important number of cases, pure, or reputed pure, animal and vegetable oils or mixtures of these, but containing no mineral oil, are used. These occur mainly in the textile trades, and while the preference for fatty oils may be due in part to belief in their superior lubricating properties, it is undoubtedly due in many cases to their superior scouring properties, and freedom from liability to produce permanent stains in textile goods on which drops may inadvertently be thrown.

2. That in all trades the use of mixed oils containing 50 per cent. and upwards of mineral oil is greatly extending, and largely exceeds the use of pure fatty oils.

3. That the use of pure mineral oils for a variety of purposes has become an important feature of the trade.

The differences which are found in practice between various oils, and particularly between mineral oils and fatty oils, the latter of which usually exhibit greater antifrictional

properties and greater power of endurance, are probably to be accounted for by differences in the molecular structure of the oils which have hitherto eluded all attempts at identification. It is possible that in the case of those mineral oils which have great viscosity at normal temperatures, and which rapidly become thinner as the temperature rises, the initial body may be due to the presence of some low members of the paraffin series, and it is probable that the greater lubricating value of animal and vegetable oils may, in part, be due to the greater constancy which they exhibit in respect to viscosity under varying conditions of temperature. But although, apart from the important element of cheapness, mineral oils offer other advantages as lubricants, e. g. freedom from acidity and from tendency to oxidise or gum, they cannot be recommended for general use in their pure state, and in other cases their use is entirely prohibited from the danger referred to above of stains occurring in fine textile goods on which drops may fall. We are therefore led to the conclusion that having regard alike to efficiency and price, the most satisfactory results are to be obtained from judicious mixtures of good mineral oil with well refined and neutral animal and (or) vegetable oil.

Body or Viscosity.—As the primary function of a lubricant is to interpose a permanent and more or less elastic film between rubbing surfaces, it is obvious that the adjustment of the body or thickness of the oil to the size of the bearing, to the weight of the machinery, to the pressure to be resisted, and to the method adopted for the application of the oil, is the final and most important consideration in the selection of a lubricant. In dealing with this aspect of the question two conditions require attention, for while it is evident that the body of the lubricant must be sufficient to withstand the tendency on the part of the journal to rupture the film of oil which is essential to successful lubrication, it has been demonstrated by Thurston, Tower and Goodman that, under uniform conditions of speed, pressure and temperature, the coefficient of friction increases, within certain limits, with

the viscosity of the lubricant. Having regard to these facts a series of experiments was undertaken by T. J. Pullin and the present writer,* the former being responsible for the mechanical, and the latter for the physical tests, in order to determine the correctness of a theory held by the observers, that the first action of a well constructed and well lubricated journal is, by the generation of heat, to reduce the body or viscosity of the lubricant to the point of least resistance, and that while the necessary temperature may vary, the viscosities of all oils at their respective working temperatures approximate to one another.

The results which are shown in the accompanying table (p. 307) prove that, with one or two exceptions, the "drag" or resistance on the bearing is controlled by the viscosity of the oil at normal temperatures, that the temperature developed on the bearing is regulated by the viscosity of the oil at normal temperatures and harmonises with the figures representing "drag," and that at the working temperature all the oils, excepting castor, have been reduced to a nearly uniform viscosity. From this the writer infers that in the case of a bearing lubricated successively with oils of different body, the temperature will continue to rise until the body or thickness of each oil has been reduced to that most suitable to the particular bearing, and until the heat generated by friction is absorbed and carried away by the oil. It is evident, therefore, that the most profitable lubricant is that which gives the lowest coefficient of friction and the lowest working temperature; but as this will probably vary with every individual bearing and with the conditions of external temperature under which it works, it follows that the selection of a lubricant is a matter to be determined rather by the discretion of each individual user than by any general rules.

But, though it is impossible to formulate arbitrary rules for the oils to be used on various classes of machinery, the following scheme, mainly applicable to compound oils, may

* 'Engineering,' April 20, 1888.

TABLE showing the viscosity of various standard lubricating oils, together with the frictional resistance or "drag," and the temperature developed by each on a $3\frac{1}{8}'' \times 8''$ bearing running at 1400 revolutions per minute = surface speed of 1100 feet with a load of 60 lbs., and the viscosity of each oil at its working temperature.

Oil.	Description.	Viscosity: Sperm = 100 at 70° F.				Normal Working.		Viscosity at Working Temperature.
		50° F.	70° F.	120° F.	212° F.	Drag.	Temperature.	
Sperm	Southern	114·6	100	67·4	53·9	18	189	56·2
Do.	Arctic	116·8	101	69·6	55·0	18·5	194	56·2
Olive	Gallipoli	218·0	177·5	85·4	58·4	26·5	202	59·5
Do.	Smyrna	240·4	188·8	87·6	58·4	25	198	57·3
Do.	Saffi	201·1	161·8	83·1	57·3	26	202	58·4
Neat's-foot	British	242·7	186·5	89·9	58·4	27	208	59·5
Lard	American	224·7	179·8	87·6	58·4	27·5	210	59·5
Tallow	British	244·9	195·5	89·9	58·4	27	204	59·5
Refined rape	Special	260·7	210·1	96·6	60·7	32	214	60·7
Do.	German	256·2	198·9	94·4	60·7	32	213	60·7
Do.	English	224·7	184·3	89·9	60·7	28	207	60·7
Castor	French 1st	..	2188·8	321·3	74·2	41	232	70·8

The viscosities in this table were determined by the old glass viscosimeter referred to on p. 285, and differ to some extent from those given elsewhere, which were determined by a viscosimeter on the Redwood principle.

be regarded as representing the limits adopted by the best oil manufacturers and by the most experienced and careful engineers in the United Kingdom.

In the following notes "S. G." = sp. gr. at 60° F. (15·5° C.).

In the case of cylinder oils, the standard for "V" = viscosity at 180° F. (82° C.) is good British tallow = 100 at 180° F.

In the case of engine and machinery oils, the standard for "V" = viscosity at 70° F. (21° C.), is finest sperm oil = 100 at 70° F.

Flashing point = "F. P." is given in all cases by "close test" in degrees F.

Steam Cylinders, Pistons and Valves.—When these work in connection with surface condensers, pure hydrocarbon oils are the only permissible lubricants, as the fatty acids which may be liberated when animal or vegetable oils are exposed to the action of steam and heat are liable to produce deposits in cylinders, condenser tubes and boilers, and to corrode the metal of these. When ordinary spray or injector condensers are used, the addition of a little refined neutral tallow, or animal oil, may be recommended, especially in the case of heavy horizontal cylinders or slide valves. Charcoal filtered oils have S. G. about 890, V. 200 to 300. Natural oils have S. G. about 900, V. 200 to 350. F. P. ought not to be under 500° F. Rate of evaporation ought not to exceed 1 per cent. after exposure for one hour to 370° F. by Archbutt's test, see p. 295.

Marine Engines.—For engines of largest size in hot climates castor oil is frequently used, especially in steamships trading to India; but cooler and cleaner bearings and greater satisfaction are said to be obtained from mixtures of heavy mineral oil and thickened vegetable oil, giving S. G. 925 to 935, V. 1000 to 1500, F. P. 350 F.

For marine engines of moderate size in temperate climates, pure animal and vegetable oils are occasionally used, e. g. lard, refined rape or olive; but compound oils as above are more commonly preferred, S. G. 920 to 930, V. 750 to 1000, F. P. 350 F.

Land Engines.—For these, according to size, pressure and speed, the limits given for marine engines may usually be followed. When circulating pumps and filters are adopted, a thinner oil may be used on account of the steady and more copious application which they provide without waste of the lubricant.

Dynamos.—For these, on account of the extreme diversity in size and speed, a great range of oil is called for—from the lightest spindle oil for the "model" in a shop window, to the heaviest marine engine or castor oil for the great dynamos for municipal lighting or for transmission of power.

Locomotives.—Refined rape oil, formerly used almost universally on locomotives, is now giving place to pale compound engine oils of moderate weight and body—S. G. 920 to 925, V. 500 to 1000, F. P. 350. Some railway companies are turning their attention to the use of natural oils, with the addition of a small percentage of refined animal or vegetable oil.

Textile Oils.—These call for consideration under two aspects: 1st as lubricants, 2nd in respect to the danger of stains which may ensue from their use. In regard to the former, olive oil, which has about double the body of sperm, may be taken as a type of the oil required for looms, shafting, and heavy (woollen) spindles; while sperm oil may be similarly taken as a standard for cotton spindles and preparatory machinery. In substituting mixed oils, containing mineral oil, for pure fatty oils, it is customary to allow 25 to 50 per cent. extra body to compensate for the lower lubricating power of mineral oil. Thus we have for looms, shafting, and heavy spindles S. G. 900 to 910, V. 200 to 300, F. P. 350; and for ordinary mule or throstle spindles S. G. 890 to 895, V. 125 to 175, F. P. 350. For ring spindles, which run in a "bath" or "well" of oil which is only cleaned out and replenished at intervals of three to six months, special oils, principally hydrocarbons, are required of low viscosity, 75 to 100, and free from any tendency to oxidise or gum, or to develop acid through long exposure to the atmosphere.

The subject of staining is too complicated for full discussion in the space at our disposal—suffice it to say that textile manufacturers are confronted by stains of three kinds:—

(*a*) Metal stains produced by drops of black oil containing abraded metal. Such drops which may be produced by the action on metal of the free fatty acids of animal or vegetable oils, usually accumulate on the picker spindle, whence they are thrown on the cloth by the picker; they are almost ineradicable, and can only, if at all, be removed by the use of oxalic or other acid, which at the same time injures the cloth.

(*b*) Primary stains due to the colouring matter present in ordinary mineral oils. This may be detected by permitting a drop of oil to fall on white cloth or white blotting paper; if the oil possesses this property it will produce a yellow or greenish mark, while fatty oils, such as sperm, neat's-foot or olive, and certain so-called stainless mineral oils, produce only a transparent colourless disc.

(*c*) Secondary stains produced by drops of oil which, though they do not immediately discolour white goods, cannot be scoured out by ordinary processes, and may produce faults in fine white goods or irregularities in dyed goods.

As hydrocarbon oils are not amenable to the action of alkalies or of acids by which fatty oils are rendered soluble in water, mineral oils cannot be removed from textile goods by the processes adopted to discharge fatty oils, but it is held by various authorities that if mineral oils are mixed with a sufficient quantity of any fatty oil to produce a complete emulsion on the addition of an alkali, the mineral oil will be taken up mechanically and discharged with the fatty oil.*

For *light high-speed machinery*, such as rotary printing presses, lathes, engineers' and machinists' tools, wood-working machinery, &c., oils of moderate body (V = 150 to 300), but containing a liberal percentage of fine animal or vegetable

* Carl. Otto Weber, Journ. Soc. Chem. Ind., June 1892, p. 495.

oil, ought to be employed. For engineers', ironfounders' and shipbuilders' tools, for which quantity is frequently more important than quality, mineral oils of heavy body (V = 1500 to 2500), with or without the addition of a little animal oil, may be recommended. For heavy bearings heated by steam, e. g. calenders and paper-drying cylinders, either a cylinder oil or a solid lubricant ought to be used.

Solidified Oil.—Under this generic name a new series of lubricants has been introduced within the last fifteen years. They have usually the consistency of palm oil or coconut oil at 60° to 70° F., and it is said that some of them do not melt under 212° F. In a few cases these oils have an opaque cream-like appearance, and consist mainly of fatty matter in combination with an alkali (potash or soda) and water and mineral oil. The greater number consist mainly of mineral oil in which is dissolved a soda or lime soap, or the materials thereof. In many cases water is entirely absent, but when present it rarely exceeds 2½ to 3 per cent. The presence of free fatty acid, which varies from 0·5 to 10 per cent., is a noteworthy feature in these oils, and must seriously affect their value as lubricants. It is claimed that solidified oils offer great economy in use from the fact that they do not flow until the bearing has developed sufficient heat to melt them; but it seems probable that the saving which may be effected in this way may be more than counterbalanced by the extra power represented by the higher temperature of the bearings, and by the danger to the bearings which the presence of acid involves.

Railway and Wagon Grease.—The first of these consists essentially of a mixture of a more or less perfectly formed soap, water, sodium carbonate, and neutral fat, and is adapted for use on the axles of locomotives, railway carriages, and trucks which are provided with grease-boxes, while the second is a soap of lime and rosin oil, with or without water, and is used on all railway trucks unprovided with axle-boxes, and for ordinary road vehicles.

The requisites for a good "locomotive grease" for high

velocities are:—(1) a suitable consistency, such that it will neither run away too rapidly, nor be too stiff to cool the axles; (2) lasting power, so that there may be as little increase of temperature as possible in the axles, even at high speeds; (3) a minimum of residue in the axle-boxes.

In practice, it is found that a grease containing 1·1 to 1·2 per cent. soda gives the best results. The process of manufacture is very simple, Morfit's soap-pans, provided with stirrers, p. 152, Figs. 35, 37, are the most suitable vessels for the purpose. The fats, usually tallow and palm oil, are heated to 180° F. (82° C.), and into them are allowed to flow the sodium carbonate and water heated to 200° F. (93½° C.); the whole is then well stirred together, and run into large tubs to cool slowly. Many railway companies buy a curd soap made from red palm oil, dissolve it in water, and add thereto enough tallow and water to bring the composition of the whole to the desired point. It is usual to allow 2½ per cent. for loss by evaporation of water during the manufacture. The composition has to be slightly varied according to the season of the year; the following formulæ for mixing have stood the test of successful experiment; the summer one ran 1200 miles.

Materials.	Summer.	Medium.	Winter.
	per cent.	per cent.	per cent.
Tallow	22	20	18
Palm oil	12	12	12
Sperm or colza oil	1	1·5	2
Soda crystals	5	5	5
Water	60	61·5	63
	100	100	100

It should be carefully borne in mind that a chemical analysis of locomotive grease is no test whatever of its practical value, which can only be determined by actual experiment in some such apparatus as has already been indicated. The instructions given for the analysis of oils

and soap (Chapters IV. and XI.) are also suitable for the analysis of products of this class, all of which contain a large amount of unsaponified oil.

The "wagon grease" is thus prepared. A good milk of lime is made, and run through several overflow tubs, where all grit is deposited; it is then drained on canvas. If the grease is to be made without water, the paste must be agitated with rosin spirit, which expels the water, and it is then thinned with a further quantity of rosin spirit. The aqueous milk of lime, or the mixture of lime and rosin spirit, is then stirred together with a suitable quantity of rosin oil in a tight barrel furnished with a shaft and stirrers, without the application of heat, after which the whole is run out into barrels to set. Many other ingredients are often stirred in, such as "dead oil," petroleum residues, graphite, seaweed jelly, sodium silicate, oil refiners' foots, micaceous ores, steatite, Irish moss, &c.

We have to express our thanks to Messrs. Baird & Tatlock, 14 Cross Street, Hatton Garden, London, E.C., to whom we are indebted for blocks appearing on pp. 274, 287 and 292, illustrating instruments of which they are the makers.

CHAPTER XIII.

CANDLES:—Raw Materials, Their Sources and Preliminary Treatment.

HISTORICAL SKETCH.

Probably the earliest known means of lighting was the torch, usually cut from pitch pine, and sticky with exuded rosin; it was used largely of old in northern countries, and even in Rome (Lat. *tæda*). Next came the link (Gr. λύχνος), a mere mass of cordage, thoroughly saturated with rosin and pitch, a source of light which even still, on foggy days, connects us with the past. After that appeared the *flambeau*, which was a finer kind of link, consisting of a centre of oakum, surrounded with alternate layers of rosin and crude beeswax.

These may be said to have formed the main illuminating agents of the upper classes up to the 16th or 17th century, the lower classes contenting themselves with rushlights, the origin of which probably dates back to the 1st century after Christ.* Etymology shows that the word candle (Lat. *candela*, Gr. κανδήλα, Persian *kandeel*, Sanscrit *kan*), is derived from roots signifying to shine or burn; it is not met with in any classical writings till towards the end of the 2nd century, and it is clear that candles were at no time considered so respectable as lamps. A chandler's apparatus was found at Herculaneum, and in the British Museum is a fragment of a huge candle (i. e. a wick surrounded with a coating of wax or tallow) supposed to have been made during the 1st century.

* For an exhaustive account of the ancient modes of lighting, and a disquisition on the etymology and nomenclature of the illuminants, the Cantor Lectures of Leopold Field, F.C.S., published in the Soc. Arts Journal, Jan.—Mar. 1883, may be consulted with advantage.

Wax and tallow continued to be used, to the exclusion of every other solid illuminant, till very near the end of the 18th century, when the sperm fishery began. Spermaceti is the best and most beautiful material for candles that exists, but its cost is a most serious objection to its use. Early in the 19th century the brilliant researches of Chevreul demonstrated the constitution of fats (pp. 4–11), and subsequently the practical genius of de Milly in France, and later of G. F. Wilson in England, applied this knowledge to the successful elaboration of the two chief industrial processes by which candles are made from stearic and palmitic acids (Chap. XV.). In the latter half of this same century, the observing insight and untiring energy of James Young created the paraffin candle, which was followed by the utilisation, for the same purpose, of ozokerit, or earth-wax, by Field.

RAW MATERIALS.

In addition to tallow, palm oil, and the various fats and oils described in Chap. III., there are several other classes of hydrocarbons which are largely used by the candle maker only; a short account will now be given of the sources and chemical constitution of these, as well as of their preliminary treatment and preparation for conversion into candles.

Waxes (including Spermaceti).

Waxes may be defined as bodies of a certain viscid plasticity when warmed, composed of fatty acids of the adipic series, $C_nH_{2n}O_2$, either free, or in combination with an alcohol radicle, or of a mixture of both. They are very difficult to saponify, yielding no glycerin, and their soaps are only sparingly soluble in water. The following list includes most of those valuable to the candle maker:—

Animal.

	Origin.
Beeswax	*Apis.*
Spermaceti	*Physeter macrocephalus.*
Chinese wax	*Coccus ceriferus.*

Vegetable.

	Origin.
Carnauba wax	*Copernicia cerifera.*
Myrtle „	*Myrica cerifera.*
Japan „	*Rhus succedanea.*
Palm „	*Ceroxylon andicola.*

Beeswax.—The composition of beeswax has been settled by the elaborate researches of Otto Hehner; the discrepancies in former analyses being probably due in great part to the examination of adulterated articles. As many as 40 different varieties are known, nearly all of them characteristic of their native lands, and although they differ greatly in physical characters, their composition (when pure) varies but little. The two chief constituents are cerotic acid, $C_{27}H_{54}O_2$, 12 per cent., and myricin $C_{46}H_{92}O_2$, 88 per cent., with traces of a substance very difficult to isolate, for which the name cerolein is proposed. When beeswax is treated with alcohol, the cerotic acid dissolves, leaving the crystalline myricin. This substance is a typical wax, being a palmitate of myricil $\left.\begin{matrix}C_{16}H_{31}\\C_{30}H_{61}\end{matrix}\right\}O_2$. The general formula of a true wax is $\left.\begin{matrix}C_nH_{2n+1}\\C_nH_{2n+1}\end{matrix}\right\}O_2$.

A considerable quantity of beeswax is imported annually from Corsica, and smaller amounts from India, Ceylon, North America, and Brazil. It is always of a brownish or yellowish colour, and has a peculiar smell resembling, and derived from, honey.

Commercial beeswax is adulterated with almost every conceivable fat, cheap paraffin, &c., as well as with "make-weights" of a coarser description; by careful sampling, a keen buyer manages to escape with about 75 per cent. of pure wax. The first operation is to boil it on weak acid water, and to separate the dross by subsidence. It is bleached either by air, or by potassium bichromate and hydrochloric acid, much as directed for palm oil, p. 83. Thus treated, however, it becomes much more crystalline, owing to the myricin being split up into palmitic and cerotic acids. When

it is air-bleached, it is melted in hot water, or by steam, in a vessel either of tinned copper or of wood, and allowed to settle; the waxy superstratum is run off while fluid into a wooden trough having a row of perforations in its bottom, by which it is distributed upon horizontal wooden cylinders, made to revolve with their lower portions surrounded by cold water. The ribbons or films made in this way are then exposed to the bleaching action of the atmosphere and the sunlight, being frequently moistened and turned over during the process. It is necessary to guard against wind, which might scatter the shreds; for this purpose, large cloths are provided. The operation is continued until the wax becomes perfectly clean and white. It is usually conducted from April till September, the exigencies of the weather preventing it at other seasons. In France, it is customary to add a little cream of tartar or alum to the water in which the wax is melted, by which the long and tedious operation of bleaching is rendered much shorter. Bleaching agents, such as chlorine, cannot be employed to bleach wax, since they render it unfit for making into candles. Purified in the above manner, beeswax is perfectly white, and has neither taste nor smell; it has a sp. gr. of from 0·960 to 0·996; at a temperature of 86° F. (30° C.) it becomes soft, and melts at 154° F. (68° C.); at 32° F. (0° C.) it is hard and brittle.

Spermaceti (Germ. *Wallrath*, Fr. *Blanc de baleine*) is written in old works *sperma ceti*, testifying to the belief then current that it was the spawn of the whale. The history of the rise of this industry is fraught with interest.* The cachalot or sperm whale (*Physeter macrocephalus*), so called from its enormous head, 14 ft. high and 25 ft. long, is the main source of this beautiful candle material. It is found not only in the "head-matter," but generally diffused throughout the blubber; moreover, it is now acknowledged that the oil from the "bottle-nose" whale (*Balæna rostrata*) contains a considerable quantity of spermaceti, of a slightly higher melting-point than from the cachalot, which melts at 113° F. (45° C.).

* Consult Field's Cantor Lectures, *loc. cit.*

Spermaceti mainly consists of cetyl palmitate, $\left.\begin{matrix}C_{16}H_{31}\\C_{16}H_{33}O\end{matrix}\right\}O$, which, on distillation, yields a peculiar substance called *ethal* (cetyl alcohol, $C_{16}H_{33}OH$), the alcohol of cetene $C_{16}H_{32}$, one of the higher terms of the olefin series, of which ethylene, or olefiant gas, is the type, and the lowest term.

The first operation needed to fit spermaceti for use is technically termed "bagging." The crude sperm oil, as brought in by the whalers, is placed in a reservoir, at the bottom of which are a number of pipes leading into long bags lined with linen, and temporarily closed at the bottom by tying cords round the mouths. The pressure exerted by the body of material in the reservoir forces a large proportion of the oil through the pores of the sacking, leaving behind the solid or "head-matter," as a dingy brown mass. This so-called crude or "bagged" sperm is deprived of a further quantity of oil by the application of pressure. It is put into hempen bags, which are deposited between the plates of a hydraulic press. The pressure applied is about 80–90 tons. When the oil ceases to flow, the sperm is taken out, melted by heat, and then drawn off into trays to granulate. The brittle crystallised blocks are ground to a coarse powder by means of revolving cylinders; the powder is collected in a bin beneath, and is filled into cloths and subjected to a hydraulic pressure of about 200 tons. The oil expressed under this force contains a small amount of solid matters, and is therefore returned for re-bagging. The blocks, as turned out of the press, are melted down, and boiled for 2–3 hours with caustic soda lye at 22° Tw. (14° B.), in the proportion of 40 parts by measure of the former to 1½ of the latter. It is important to guard against an excess of alkali beyond that required for combination with the oil, as it would tend to saponify the spermaceti, and cause a waste of material. The mixture is kept at a low equable temperature, till the oil is taken up, and is allowed to remain at a gentle simmer, while the soap that has been formed rises to the surface and is skimmed off. The heat is then raised to about

250° F. (121° C.), and the mass is treated with small successive doses of water, the additional scum being carefully taken off as it rises, till the whole is clear. It is then drawn off to crystallise in flat tin dishes, whereupon the cakes are again reduced to powder, placed in linen bags, and subjected to a hot pressure in a very powerful hydraulic press heated by steam, after which the spermaceti will still contain a quantity of oil, or weak sperm, which no more pressure will remove, and which must be extracted by saponification. The final operation consists in boiling down the sperm with strong potash lye at 235° F. (112° C.), removing the scum as before. When the latter ceases to appear, further purification is effected by introducing a little water, at intervals, while the heat is lowered. The supernatant spermaceti, now perfectly colourless and transparent, is cast into blocks and crystallised.

Spermaceti candles are valued for their beauty and illuminating power. They usually contain about 3 per cent. of wax or paraffin, to counteract the crystalline structure of the spermaceti, and are moulded in hand frames (see Chap. XV.), and are chiefly used for photometric purposes. The addition of a little gamboge makes the spermaceti resemble wax, the compound being known as "transparent wax."

Chinese Wax (Chung peh-la) is produced by a small insect like a woodlouse, the *Coccus ceriferus*, whose cultivation is now an industry next to silk in importance. According to Thistleton Dyer, F.R.S., whose position at Kew Gardens affords him exceptional facilities for investigating questions of economic botany, the annual value of the amount produced is computed at about 600,000*l*. The statements as to the tree on which it feeds, and whose branches it covers with wax, are conflicting. *Fraxinus chinensis*, however, is certainly cultivated for the purpose. The wax is a cerotic ether (ceryl cerotate), $\left.\begin{matrix}C_{27}H_{53}O\\C_{27}H_{53}\end{matrix}\right\}O$, corresponding to the true cerotic ether $\left.\begin{matrix}C_{27}H_{53}O\\C_2H_5\end{matrix}\right\}O$, or ethel cerotate. It melts at

180° F. (82° C.), and can be crystallised unchanged from boiling alcohol. It has a longitudinal crystalline fibre, like stearin or stearic acid, and yet possesses some of the flaky qualities of sperm.

VEGETABLE WAXES.—*Carnauba wax*, or stone wax, is found in thin films on the leaves, stalks, and berries of a Brazilian palm, *Copernicia cerifera*. Its sp. gr. is 0·999, and its melting-point 185° F. (84° C.); the composition of it is not definitely settled, but according to Allen it contains a notable quantity of free myricyl or melissic alcohol $\left.\begin{matrix}C_{30}H_{61}\\H\end{matrix}\right\}O$. Lewy says that it contains 80 per cent. of carbon. Stürke * states that he has succeeded in isolating from it seven distinct bodies—one a hydrocarbon melting at 139° F. (59·5° C.); three alcohols, one of which melts at 218° F. (103·6° C.); and three acids, one of which is identical with cerotic acid.

Japan wax is obtained by boiling the berries of several trees of the genus *Rhus*, from incisions in the stems of which flows the famous Japan lacquer varnish. It ought properly to rank as a fat, for it consists almost entirely of glycerin palmitate, and yields glycerin upon saponification. Its sp. gr. is 0·984–93, and its melting-point 120° F. (49° C.). It is largely used in vegetable-wax candles, which are made as a substitute for beeswax.

There are several other waxes, much used in their native countries, as Palm wax, from the stem of *Ceroxylon andicola*, and Ocuba wax, from *Myristica bicuhiba*, both growing in Brazil, and furnishing most of the candle power of northern South America; also Andaquies wax, Cuba wax, and others of uncertain origin.

PARAFFINS.

To Dr. James Young belongs the honour of creating the paraffin industry. In 1848 his attention was called by Dr. Lyon Playfair to a little stream of oil perpetually

* Jour. Soc. Chem. Ind., iii. 448.

running in a small garden on the top of a coal seam at Alfreton, in Derbyshire. From this oil he extracted paraffin, a light burning oil, and heavy lubricating grease. In 1850 he took out his celebrated patent for the distillation of coal minerals at a temperature not exceeding 600° F. (315° C.). Just prior to Dr. Young's invention, Rees Reece had obtained paraffin crystals and liquid, by distilling slowly the tar which resulted from the rapid distillation of peat. Since 1862, the bituminous shale which abounds in Linlithgow and Midlothian has been the only source of paraffin oil, of which 22–38 gal. are obtained from a ton of shale. This crude oil is, after distillation, washed with acid and soda tars returned from the later stages of the oil refining process, and distilled again. The oil from this second distillation is known as green oil, and is passed through the refrigerator and cooled to about 36° F. (2° C.) and then pumped through a filter-press. The separated green pasty mass is then subjected to heavy pressure in the hydraulic press, and the cake so obtained is known as "hard scale." The mother liquor from these pressings is, after another treatment with 2 per cent. of strong sulphuric acid 1·845 sp. gr., and 1½ per cent. of caustic soda solution 1·36 sp. gr., again distilled. This distillate is mixed with the intermediate oils "·860–65," from the collateral distillation of a lighter fraction, cooled to 18° F. (– 8° C.), sent through the filter press, and afterwards through the hydraulic press, as in the former case. The solid product of these operations is known as "soft scale."

Paraffin scale testing.—This commodity is of somewhat variable composition. The impurities amount, on an average, to 10–12 per cent. of the weight, and consist of blue oil, greasy hydrocarbons of low fusing-point, solid refuse, and water. The oil manufacturers, having an eye to the quantity of solid product, often separate the scales from the oil as early in the process of oil refining as possible, knowing that the subsequent distillation and the treatment with acid and alkali will reduce the weight of solids by decomposition.

To the practised eye of the buyer, deficiency of quality arising from lack of treatment is at once evident, and determines the general character of the scale, but minute differences arising from insufficient pressing can only be estimated by testing. A method of testing has been agreed to between the producers of Scotch scale and the buyers, for determining (1) the amount of water, (2) the amount of dirt, and (3) the excess of oil; and deductions are made off the invoice weight or price, for all excess of dirt and water over 2 per cent., and for all oil expressed over 4 per cent., 6 per cent. of impurities being taken as the normal amount.

The sample is taken by means of a long, slightly conical, metal tube. The smaller end of the sampling tool is inserted through a hole bored for the purpose in the end of the cask, and driven, auger fashion, right through its contents. The sample of the lot having been mixed, is put into wide-mouthed bottles with glass stoppers, or with good corks, which have been previously saturated with paraffin wax. It is important that these should be quite full, as otherwise partial evaporation may occur, and moisture may condense in the upper portion of the bottles.

For the estimation of the dirt or insoluble matter, which will be found to consist of fibres from the press cloths, and particles of sand or mud accidentally introduced, a fairly large sample is recommended to be taken: where convenient, as much as 7000 *grains* (453·58 *grms.*). This is weighed and melted, and after subsidence the clear portion is poured off. The residue, after washing with gasolene, is poured on a tared filter, and after further washing, dried and weighed. The result, divided by 70, will give the percentage of "insoluble."

The water is determined by distilling a pound of the sample in a copper flask with Liebig condenser attached. The flask is heated by a Bunsen flame, and the distillate is collected into a narrow graduated measure. A little water may be found adhering to the sides of the condenser tube. It must be rinsed out with a little gasolene, and added to the distillate in the flask; the volume of the water may now be

read off, and the percentage, in this instance, got by dividing the number of measured grains by 70. Another method, adopted at Price's Patent Candle Co.'s works, is as follows. "500 grains of the scale to be tested are weighed in a tared porcelain basin, and heated with constant stirring to 230° F. (110° C.), until bubbles cease to be given off; the loss is then determined. 500 grains of the same scale, which has been freed of its water and dirt by melting at a gentle heat and subsidence, are to be treated in the same way, and the loss again determined. The loss in the second instance is now to be deducted from the loss found in the first, and the remainder is then to be taken as the quantity of water present." This divided by 5 will give the water percentage.

For the oil test the portion of the clear wax poured off in the process for the determination of the insoluble may be put in a basin, and cooled down as slowly as possible overnight to 60° F. (15·5° C.), the temperature at which the operation of pressing has to be conducted. This is done in a room where the temperature can be regulated. Any kind of press may be used for the test, provided the following conditions are fulfilled. The area of pressure must be 20 square inches, and there must be a means of measuring the pressure used. The pressure agreed on is 10 cwt. per inch super, the temperature 60° F. (15·5° C.), and the duration of pressure 15 minutes. A very compact press on the hydraulic principle, which has been very generally adopted by the trade, is shown in Fig. 85. It is made by Clarkson and Beckitt, of Maryhill, Glasgow, and was designed by the writer, jointly with the late Alexander Smith, draughtsman to that firm. It is wrought in preference with oil, and the oil cistern and cylinder are in one piece, which forms the press support. A Bourdon gauge indicates the pressure, and a small brass tube screwed slantingly into each of the cast metal saucers forms a mercury reservoir, into which a small thermometer can be placed to indicate the temperature. The horizontal sectional area of the ram is 20 square inches, and the pressure area in each of the four saucers, which are turned on both sides, is

the same. One of these is represented lying on the chair. The bottom of each saucer is so reduced as exactly to fit the inside of the one immediately below. Four samples can thus, if required, be pressed at one time. The sample is now pounded in a mortar, and a quantity of 250 *grains* (16·2 *grms.*) in the case of hard scale, or 150 *grains* (9·6 *grms.*) in the case of soft, are carefully weighed. Six discs of strong filter paper are now laid in the bottom of the saucer, and over

FIG. 85.—SCALE TESTING PRESS.

them one of fine linen. On this the weighed scale is carefully spread, and then covered with another linen disc, and six more discs of paper. When the thermometer fixed in the press saucer indicates 60° F. (15·5° C.), the pump of the press is put in action, and continued till the gauge points at 10 cwt., which is equivalent to 10 tons on the area of the cake. This pressure must not be exceeded. The press may now be locked, and allowed to stand at this for 15 minutes.

When this time has elapsed the press is unlocked, the ram is forced back into the cylinder by means of the screw actuated by wheel lever, and the oil, which is used by preference in this press, is sent back to its reservoir. The saucers containing the pressed cakes are taken out, and the cakes, having been carefully removed from their coverings, are weighed, and the loss noted. As the dirt and water have been previously removed, the result has to be corrected. Supposing that in this case the 250 *grains* pressed represented a scale known to have contained 2 per cent. of dirt and water, and the loss by pressure is 10 *grains*—

$$250 : 98 :: 10 : x = 3 \cdot 92,$$

the report will therefore be for a hard scale—

	Per cent.
Dirt and water	2·00
Oil expressed	3·92
Clean scale	94·08
	100·00

With Scotch scale at least, pressed in its original state, it has been found that a portion of the water is pressed out along with the oil, and a correction had to be made by deducting this quantity, which had to be determined otherwise from the loss by pressure, but this necessity is done away with in the process detailed above, where the scale pressed has been previously deprived of its moisture.

The "setting point," commonly called the "melting point," is determined by what is known as the English test. A test tube, about an inch in diameter, is filled to a depth of two inches with the melted scale. A thermometer is inserted and the whole steadily stirred. When congelation begins to take place the mercury will remain for a short time stationary. This point is noted as the setting point of the sample. Scale setting below 118° F. (48° C.) is classed as soft scale, and a money allowance for each degree F. that the scale is lower in melting point than this is often stipulated for in contracts.

It is not to be understood that the loss by pressure is in any sense a measure of the oil existing in the original scale. The test merely furnishes a basis on which buying and selling can be effected. By the rejection by the buyer of delivered lots, the seller, previously to the adoption of this test, was sometimes put to very serious trouble and expense; and, on the other hand, during the first year of its adoption, one firm, to the writer's knowledge, received in allowances over 800*l.*, which was about 3½ per cent. on the total purchases, and of which the greater portion would have been loss in the absence of the test.

American scale is much freer from impurities of all kinds. In working it will be found much more homogeneous, containing a shorter range of melting point. It is now much easier refined, and in some cases, for cheap candles, it can be freely mixed with semi-refined, a procedure which, in the case of Scotch scale, is entirely out of the question.

The process of refining may vary according to circumstances. The method adopted by Young and by Walls and Co., of Glasgow, is first to melt the scales in a large pan, by the introduction of steam through a perforated wrought-iron coil. The mechanical impurities and the bulk of the water subside; the supernatant liquid is decanted into another vessel, and mixed with a due proportion of shale or spirit, varying in gravity from 0·735 to 0·765. It is then caked, or allowed to fall on a revolving drum, kept cold by an internal flow of water; less spirit—say ¼ of the weight of the scales—is required by the former plan, more by the latter. The cakes or pulp, as the case may be, are then placed between porous or absorbent materials, usually cocoanut matting lined with canvas. These are arranged in a hydraulic press, fitted with iron plates and wickerwork mats to keep the layers distinct, and are subjected to pressure. This operation is repeated till all the oil and greasy hydrocarbons are removed. When this is accomplished, the cakes, as removed from the press, should be almost transparent. Instead of employing clean shale spirit at each operation, some manufacturers,

with a view to economy, use the pure liquid for the last wash only. When this principle is adopted, the liquid pressed from the cakes at the last operation is used for the second melting, and the pressing from that stage again for the first melting. Thus the scale is subjected to 3 pressings, which, if done with care, should be sufficient to produce highly refined paraffin. It is obvious, however, that unless the scale has been properly treated by the oil refiners, before coming under the operations described, disappointment and failure will result. It is also clear that the refining will be more perfect by using pure spirit at each operation.

The oldest form of hydraulic press was the vertical cold press, still used for the commoner kinds of fat. The newly introduced hot presses, on the other hand, are horizontal and altogether of superior construction. They are made by Needham and Kite, of Vauxhall; and by Galabrun Frères, of Paris. Figs. 86, 87, and 88 show respectively a front elevation, partly in section, side elevation, partly in section, and a plan, of a novel hot-press specially adapted to the requirements of paraffin, sperm, and stearin refiners. It is made by Clarkson and Beckitt, Maryhill Engine Works, Glasgow, from the design of the late W. Walls, of that city, through whose kindness the following description and illustrations were obtained. For convenience in working, and economy of labour, whether as a hot or as a cold press, it is unequalled. The plates A are notched at each end with a check of varying width, corresponding with the width of 2 vertically tapered bars B, placed at each end of the press and parallel with its supporting pillars. The object of this contrivance is to do away with the necessity for a balance weight, or other arrangement for bringing back the ram, plates, &c., when the press is opened. By this plan, on the ram being lowered by its own weight, each plate falls back into place, being stopped by the increasing size of the tapered bars; thus the plates are held in their places, equidistant from each other, and in a position to render the refilling of the press a matter of the greatest ease and

simplicity. The bottom of the press is cast with a trough on it, which receives all the drippings from the plates, while the cylinder head, and the bottom collars on the pillars, are provided with projecting drooping rings; in this way no oil can escape down the sides of the tapered bars or the pillars, but all must drop into the trough. The press is entirely encased in sheet iron, with hinged doors in front, for introducing and withdrawing the materials. A perforated steam pipe C is introduced at the top of the press, and is carried down the back inside; thence it passes on both sides around the bottom of the trough, for the purpose of keeping the drippings in a liquid state. An outlet pipe is attached to the front or back of the trough, and serves to conduct the oil to a tank.

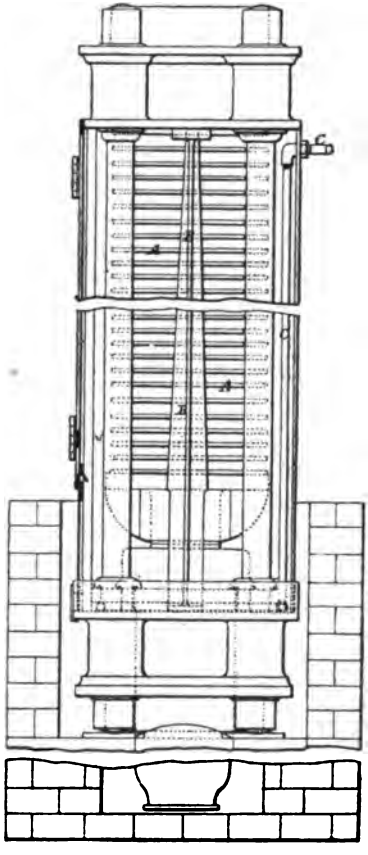
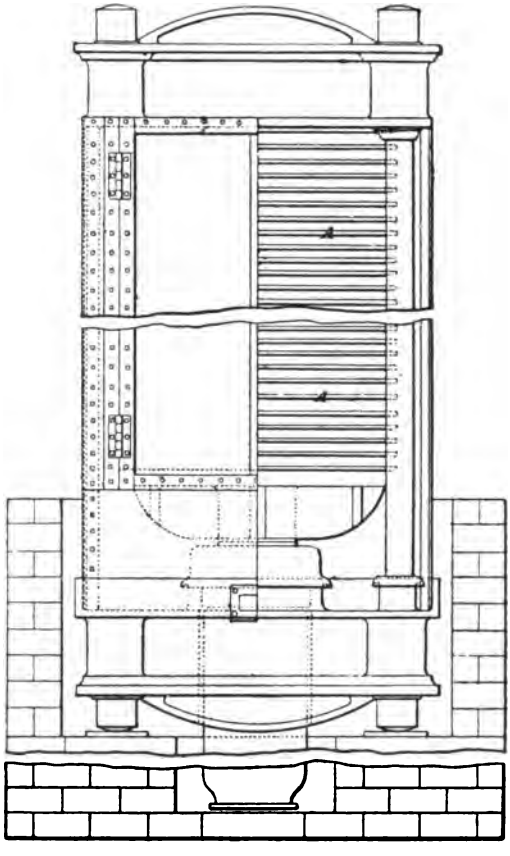
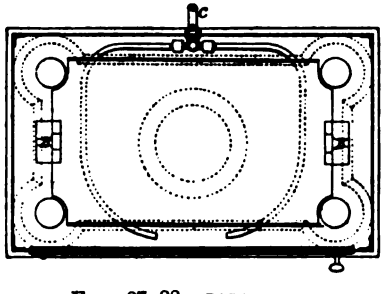


FIG. 86.—PARAFFIN PRESS.

This process of purification is simply mechanical; its efficacy, other things being equal, may be a subject of arithmetical calculation, the factors being the relative quantity of spirit used at each wash, the amount of oil and spirit left in at each pressure, and the number of washes. There is, moreover, another matter which exercises an important





FIGS. 87, 88.—PARAFFIN PRESS.

bearing upon the results, viz. the temperature at which the cakes are cooled after each melting. In the act of cooling, crystallisation takes place, and the slower this is performed the more perfect are the crystals. The result is that when a perfectly crystallised cake is subjected to pressure, it behaves like a sponge, yielding up the impurities to combination with the spirit. A quickly cooled cake, on the other hand, may be compared to a piece of putty or dough, and will not press to advantage, nor yield satisfactory results. The washes containing the impurities, along with paraffin in solution, are, as has been shown, either sent back in the process in bulk, or the naphtha may be distilled off, and the residue returned and treated *ab initio*. In all cases, the liquid which has been used for the washing at the first stage is the most heavily charged with low hydrocarbons, and must be subjected to distillation. The spirit is separated from this residue by steam heat, and may be used again; but the heavier portions require treatment with acid and alkali, as in the case of crude oils.

The cakes, which, by repeated washing and pressing, have been freed from colour, and from the greasy constituents which would by their decomposition injure the wax, will, even after extreme pressure in hydraulic presses heated by steam, still contain a proportion of naphtha. To get rid of this, the cakes are put into a still or rectifier, an iron vessel furnished with a condensing apparatus. Amongst the melted paraffin, high-pressure steam is introduced through a perforated pipe, and produces violent agitation. This is continued till the last trace of smell is removed, 6–18 hours being required to effect it, according to the specific weight of the oil or spirit used, and the heat of the steam. The condenser is employed for the purpose of recovering the naphtha for future use.

The only operation now requiring explanation is the passing of the liquid wax through animal charcoal. Before this process can be applied with effect, every trace of suspended moisture must be removed; this is done by

heating the liquid, as decanted from the rectifier, in a steam-jacketed pan. After the complete removal of the water, freshly burned animal charcoal, to the amount of 2½–5 per cent., is added to the wax, stirred actively for ½ hour, and left to settle. The clear liquid is then allowed to percolate through filters, which effectually remove the very fine particles of charcoal that refuse to subside. This liquid, which should now be as pure as water, is put into moulds to cool, when it will be ready for candle making.

For quality of product the spirit process is unequalled, and spirit-refined wax still commands the top price in the market. The method has, however, been superseded in great measure by another which has not the drawbacks of serious loss and risk of fire which accompanied the use of a solvent so volatile and inflammable.

A discovery arising from so slight a cause as the accidental falling of a piece of scale on a steam pipe revolutionised the method. There was no violent explosion, but it was observed that there was a change of colour—a whitening of the scale, and the reason why was sought and found. The chemist had, before this, separated, by elaborate distillations the various members of the paraffin series, and found that as the boiling point rose as the paraffins became more complex, so did the melting points. It was left to the practical man to utilise this last property as a method of refining, and of differentiating his products. He saw the liquation or sweating process in embryo in the piece of whitened wax; and a patent was taken out in 1871. On the expiry of this, Hodges' patent, in 1885, its principle was almost universally adopted by refiners; and it may be assumed that the so-called "spirit-refined wax" of to-day, is wax which has had the bulk of its impurities removed by the liquation process to be described, and finished with a wash of naphtha.

The sweating process.—As generally carried out, it is as follows:—

The scale is melted by open steam. Where by the nature of the ground it is possible to have it delivered at a high

level, the melting may be done in an open tank, and the liquid conveyed, by the force of gravitation, through a pipe to the cooling room. The advantage is great, inasmuch as the settling out of the water and dirt can be done in the same vessel. This is seldom the case, and pumping has to be resorted to in most cases. A most convenient pump for waxes, made by Clarkson and Beckitt, Maryhill, Glasgow, is shown in Fig. 89.

The pump, however, may be dispensed with at this stage if a strong cylindrical boiler with manhole and cover can be got. The boiler is provided with a hopper, the opening in the

FIG. 89.—PUMP FOR WAX.

bottom of which is tightly riveted about the manhole, which, when the charging is completed, is closed by a door of usual construction. It is the better method to have the manhole to open upwards. For convenience of charging, the boiler should be so set that the top of the hopper should be flush with the floor, which should be well fitted round its edge. Three pipes are inserted into the top of the boiler, at the opposite end from the manhole, two for letting in steam, and the third to convey away the wax after it is melted. The boiling is done by the pipe which is carried down near to, and around the bottom of the tank, throughout its length. All this lower part is

perforated with small holes. The branch which merely enters the boiler, and the pipe which extends to near its bottom, and has its end protected by a cage, are for the purpose of discharging the contents. The action is the same as the chemist's wash bottle, pressure of steam on the surface of the contained liquid overcomes the atmospheric pressure in exit pipe, and the material escapes to be conducted to elevated settling tanks. If an air engine is at command, the blower must be an independent pipe connected to the air pump. The action in both cases is the same. To fill, the manhole is opened. When the melting is finished, and

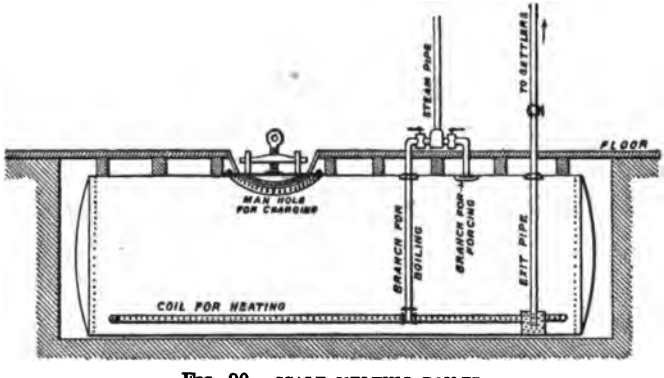


FIG. 90.—SCALE MELTING BOILER.

the mass brought to a sharp boil, the manhole cover is screwed down, and the steam shut off the coil. The short steam inlet, or pipe from air pump, and exit pipes are now opened and discharge takes place. The air pump is the better arrangement, and can be used wherever necessary throughout the factory ; all that is needed, in addition, being a pipe from air engine, and a close vessel to blow from. It is of service especially in circumstances in which water from the condensed steam is objectionable. The tanks to which the melted scale is conveyed should be near the cooling room. They should be made of iron, and this applies to all the vessels used in the process where the hydrocarbons only are

dealt with. Each tank also should have a perforated pipe in its bottom for heating up when required. Here the liquid is allowed to rest till the water and dirt have settled down and have been run off into the separators.

In the cooling room are several ranges of double racks. These may be set in pairs. Each rack is 17 in. wide, and two when set at proper distance will occupy 4 ft. width of floor space. There should be a working passage, and then a double rack again, and they may extend in length nearly the entire length of the room. It is better to set aside some of these for the wax in the more advanced stage, and others, for the cruder wax, for reasons which are obvious; nevertheless each tank should by pipes and valves be so connected with the others and the system, that the contents of any tank can be sent to any rack should occasion require.

Directly over the middle 1 ft. 2 in. space which separates the parts of each double rack, is placed the main connected to the settlers. It may be made of 4-ft. lengths of 2-in. malleable iron steam pipe, connected by couplings carrying ½-inch branches pointing upwards. On these are screwed three-way gland-stuffed brass cocks. The inlet is through the key, and the outlets point sideways. To each of the side branches is coupled a tee piece of ½-in. malleable pipe, so that when placed over the rack containing the moulds, two moulds can be filled from each branch on either side; and as the moulds are arranged in a zigzag fashion, 12 deep, there is only one valve required for 48 moulds. Each mould, except that nearest the ground, has two outlets on one side, near the lip, and after it is filled up to this, the liquid overflows into the next below, and so on to the rest. The racks may be made of angle iron, the uprights at 4 ft. distances, and the bars on which the moulds rest 4 in. apart. Each mould is 22½ in. by 22½ in. by 2½ in. of tinned steel, stamped into shape, weighs 9 lb., contains 20 lb. of wax, and costs 3*s.* 3*d.* Those referred to were supplied by John George & Son, Great Alie Street, E. It is important that the racks should be so constructed that they can be kept perfectly

level. The exact position of valves on main pipe is obvious. They should be exactly equidistant from the uprights which support the cross pieces on which moulds rest. A small steam pipe with valve is let into the mains at their filling ends; and the mains are contracted at the opposite end to ½ inch, and a valve placed there also, and the end of the small pipe bent downwards. These are for the purpose of heating the pipes previous to, and draining them after, use. This arrangement is sufficient for crude wax, but for harder, which we have to deal with further on in the process, a small steam pipe enclosed in the wax pipe is required to keep it hot. Pockets in which the wax can lodge must be avoided; and this is the reason why the branches are made to point upwards. One man can by this arrangement superintend the moulding of many tons of wax with very little effort. The wax will be ready for the next operation the next day.

A much more simple and inexpensive method, so far as plant is concerned, is also adopted, but it requires more floor space. The melted scale is allowed to fall into a shallow trough, so placed that its overflow is just about level with the surface of a large water trough, say up to 60 ft. long by 10 feet wide. The wax flows from this trough, over the water in a steady wave, till the whole sheet is covered. The thickness of the cake is determined by the quantity of the material; and in this respect absolute uniformity is secured. This, when cold, is first cut into slabs: a man with a gauge like a tee square, and a knife fixed in the blade, makes a slight incision along the trough at the required distance from the edge. Another following, by pressing sharply with both hands along the outer edge, breaks the piece off from the main sheet, and then with a gauge, measures each cake, and makes cuts across the slab throughout its entire length, so that it is easily broken up into cakes of the required uniform size. The cutting up of the rest is done in the same way, with expedition, and the cakes are removed to the hot room. One or more coolers may be placed over the first, but owing to the difficulty of supporting great weights over large

surfaces, without intermediate supports, this plan is not to be commended with large coolers, as the supports interfere with the management of the sheets of wax, which must be free to be floated at will.

The next operation is the sweating. This is done either in small cupboards in which each shelf is hollow, and a steam case—such an apparatus was patented by Appleby in 1886—or in large apartments heated by steam pipes. In either case, under the cake, a wicker frame or coconut matting is laid. In Appleby's cupboard, steam heat, never under 212° F. (100° C.) is brought into close proximity to a cake which liquefies at a temperature very much lower. Each shelf has a valve-governed steam inlet and outlet to itself, and the utmost vigilance is required to secure good sweating and at the same time prevent the cake from vanishing altogether. The expense of construction is great, considering the quantity turned out. A more rational medium for conveying heat would be water, but with steam even, notwithstanding this drawback, good work has been done, and in very short time. Cupboards of this description, with doors close up to the shelves, may be arranged in the cooling room parallel and adjacent to the cooling racks, and the use of the barrow dispensed with. The same arrangement is practicable with plain shelved cupboards, the heat in which is raised by means of steam pipes below; but when the arrangements for cooling are such as have been described at length, a separate apartment is required for the sweating process.

A good plan of erection is to have chambers, with passages in the middle throughout their length, the racks on each side, sufficient room being left behind them to allow free circulation of hot air around the shelves. The heating pipes may be disposed under the racks, as well as under the passages. The inlets should have valves, and if the steam is taken from the boiler there should also be reducing valves, as owing to irregular demands on the boiler, the supply will be unsteady. There should also be steam traps at the exhaust ends for economy's sake. The steam from the engine exhaust can be used with

advantage for this purpose, as little pressure is required. It is an advantage to have the pipes as low as possible, and as there will be, despite the utmost care, some leakage from the shelves and gutters, the floor should be made very secure and unabsorbent. The angle-iron racks on each side support shelves ranged above each other at 6 or 7-inch distances. The height to which they may be carried is limited only by the difficulty of charging them when beyond a man's easy reach. The shelves may be economically made of sheets of corrugated galvanised iron, of say 8 feet long and 4 inches wider than the cakes of wax to be laid flat on them. There are uprights of angle iron at back and front, at distances of about 4 feet, carrying cross-pieces at 7 inches apart, a little longer than the width of the sheets, to allow of their attachment to the uprights. Each sheet is thus supported at each end and at the middle. The sheets may be laid level from back to front, but there should be a dip of 2 inches in their length, and at the position where they approach each other at the low ends a gutter closed at both ends, but furnished with a drop pipe, is placed. At this part the one upright carries a double cross-piece, so that the ends of both shelves are supported, and at the same time an opening is secured for the keeping of the gutter clear. Each pair of sheets is thus provided, and each gutter conveys the drippings to its neighbour below, and eventually it is all caught into a main leading to a series of sunk tanks arranged for keeping separate the drainings of various melting points, and provided, as in the case of the boiler where the first melting is done, with an arrangement for elevating and sending to settling tanks the material for remoulding or for hydraulic pressing. Under the cakes, as described elsewhere, there is coconut matting, and to save the workmen's hands, jute canvas next the cakes. As the chamber gets heated, the cakes soften throughout. The bleaching is first apparent at the edges, then the surface assumes a spongy consistence, and on breaking a small piece off it will be seen that the brown oil is sinking through its substance by its own weight as the pro-

cess goes on. When about two-fifths of its weight has been removed, sooner in the case of good high-melting-point scale, what remains on the shelves will be found white throughout and firm. The steam is now shut off, and when the draining has ceased the shelves are emptied by manual labour. The white cakes are emptied from the barrows into a sunk tank for melting, previous to being elevated for charcoal treatment. Some water will have collected in this tank during the melting, and the pump, if not set very low, may refuse to lift this while hot. A swing pipe attached to the suction will, however, give the means of taking the wax off the top first, and the water will follow when it has cooled a little.

By an arrangement made by N. M. Henderson, manual labour is still further economised. Attendance is only required for the regulation of the process, the turning of a screw, the opening and shutting of a few cocks and steam valves being all else that is needed. This apparatus was patented in 1887. The room for moulding and draining is one: the shutting of the doors, and turning on the steam, converts a cooling room into a draining one.

In the modification of Henderson's, to be described, the houses are in pairs, divided by a 14-inch brick wall, and provision is made for another pair in a second story. The single apartment is 52 feet long and 13 feet 3 inches wide. It is 13 feet 3 inches in height from the foundation to the beams across the house, which support transverse arches. The room has large doors at each end, and is heated up by systems of steam pipes along the side walls. On two huge iron frameworks are supported two sets of frames of nine trays each, piled above each other. These trays are 21 feet long, 6 feet 6 inches wide, and 6 inches deep. They are supported at 3-feet distances along their length by six pairs of strong iron uprights, 9 feet high, carrying cross-pieces of steam tubing. The transverse pieces are fixed at 10-inch distances. The ends of the sets of trays when level are 2 feet distance from each other, and an equal distance from each door. From the top of one tray of the set, to the bottom

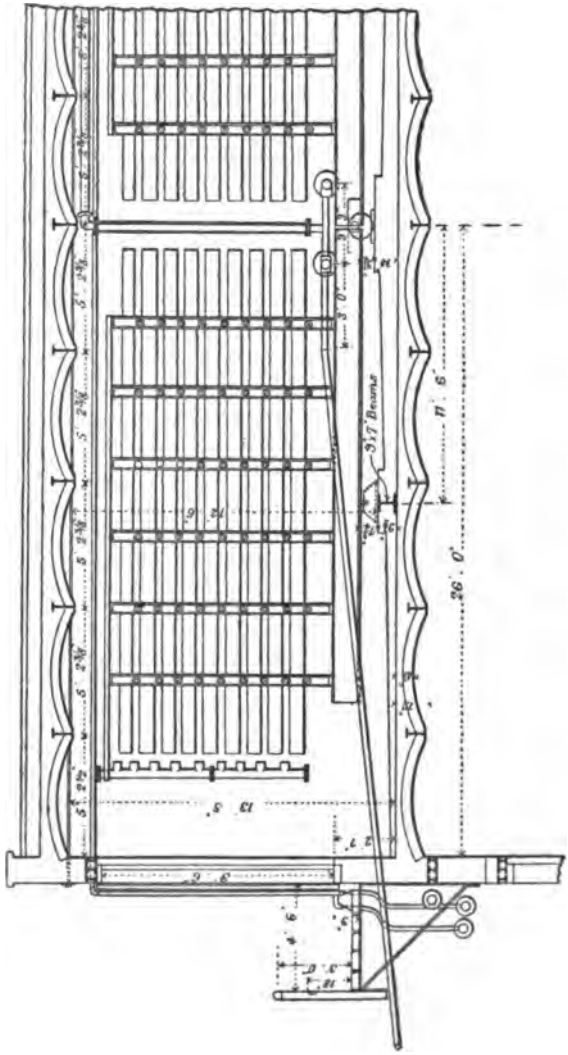


FIG. 91.—HENDERSON'S SWEATING ARRANGEMENT.

of that immediately above it, is an open space of 4 inches. Covering the bottom of each tray there are frames of galvanised wire close netting supported by fillets along the sides and the bottom. On the side near its bottom and near the corner of each tray of both sets where they approach each other, is fitted an elbow discharge-piece which can be turned in different positions. A small pinion is so attached to each elbow-piece of each set, that a rod with nine short worm-screws geared into the pinions, raises or depresses the elbow-pieces, closing and opening the trays of each set by the one movement. The special feature of the apparatus is this:—Each of the two sets of trays is balanced on a compound lever, which is composed of two parallel beams. The fulcrums are each 14 feet 6 inches from each door, and 11 feet 6 inches from the middle of the house, and are directly under the middle of the rack of trays, but the heavy iron beams on which they rest, being each 20 feet long, are made nearly to meet in the middle of the house, and thus 32 inches of lever length determines the preponderance in the direction of the middle of the house. These, the long ends of the levers, are rested on an eccentric, which, worked by a lever, sets the trays of both sets either level, or 6 inches lower when required. The points of suspension are 18 inches above the ground, and the door level is flush with top of supporting lever when it is at the level.

The elbow outlet pieces of the trays deliver into an upright pipe with nine openings, a compound funnel in fact, and both pipes are connected into another which leads to the tanks. At the opposite ends of the framework, each set has an upright pipe with nine branches and valves for filling the trays.

The method of working is as follows:—The 18 trays are set level by one turn of the eccentric which works the levers. The trays are all closed by working the worm screw of each set. Water is let in to cover the wire netting at bottom of trays, and then the melted scale to the amount of 16 tons is run in. If the weather is warm the doors are opened, and the

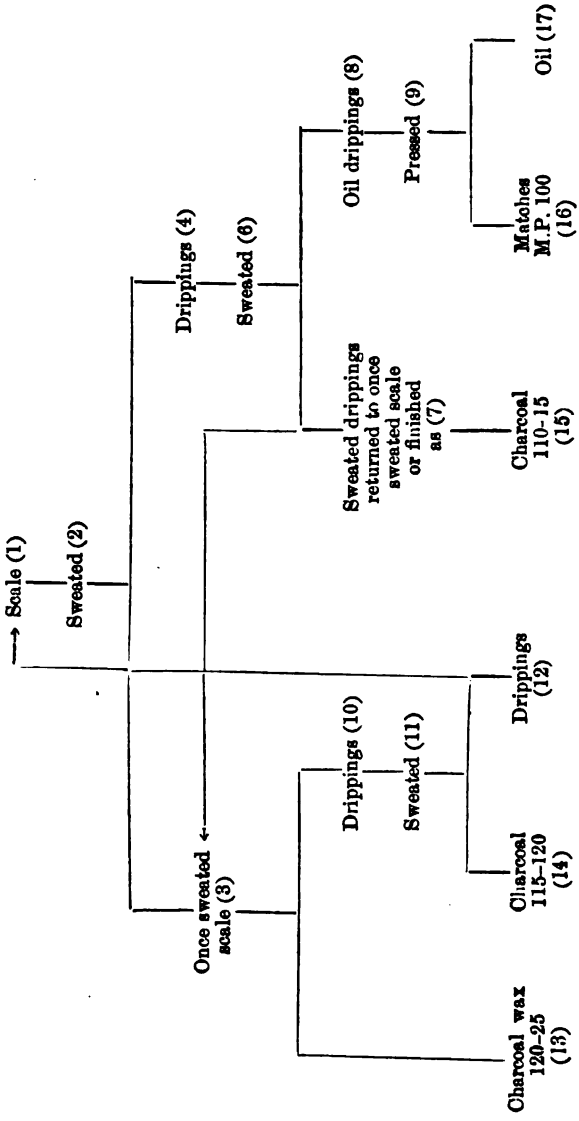
cooling operation commences. In ordinary weather, 24 hours will be required to do the cooling. When the scale has cooled and become firm, the water, by the turning down of the elbow pieces, escapes into its tank, and the sets of trays are inclined towards each other by turning the eccentric. The withdrawal of the water has left draining space below the cakes, which now rest on the wire netting. The doors are shut, and the steam turned on. The sweating may occupy 24 to 30 hours, and the drainings are diverted to boilers at a low level. The drainings below 65° F. (18° C.) setting point are returned to oil refinery, dispensing in this instance with the use of the hydraulic press. If finished wax is wanted of 118°–120° F. (48°–49° C.), the scale is diluted previously to caking by residues from previous lots to 108°–110° F. (42°–43° C.). If 125°–130° F. (52°–54° C.), then the original setting point is made 115°–120° F. (46°–49° C.), and so on, an allowance of 10°–12° F. being made for the rise of setting point during the operation. Five per cent. is usually sweated out for each degree F. that the wax is raised. By means of three-way cocks the sweatings are diverted into the various tanks; this for the purpose of being able to make different qualities from all the houses at one time. In ordinary weather each house containing two racks will turn out from 12 to 15 tons finished wax per week.

The wax left in the trays is in its turn melted out by free steam, and sent forward to charcoal treatment. Thus the disagreeable work of emptying is also avoided, which, as in the case of charging in the ordinary way with cakes, involves the exposure of the workers to more than tropical heat, causing them to sweat as well as the material.

As the requirements vary, so must the process be varied. The diagram on p. 342, by Tervet, will give an idea of the process as generally done.

The disadvantages which present themselves to the mind are, the expense of erection, the difficulty of getting the centre of the cakes which are further from the source of heat so well done as the edges, and principally, the loss of heat

DIAGRAM ILLUSTRATING THE SWEATING PROCESS.



and time arising from the use of one house for purposes so opposite. This to some extent at least is considered to be the case, but on the whole, by those who have adopted the method, it is well spoken of.

Paraffin wax finished with animal charcoal and filtering immediately after sweating, is still liable to contain some hydrocarbons of lower grades, which on exposure to the influence of light and air, develop colour which is not apparent at the time of finishing. To destroy these, the following treatment is sometimes adopted. The wax from the sweating house is melted, and heated in a still to 350° F. (177° C.). It is then turned over into a lead-lined pan, and a small percentage of sulphuric acid (1·845 sp. gr.), is added, the amount varying according to circumstances. The air agitator is turned on, and agitation is kept up briskly for an hour. After the air has been shut off, the paraffin remains at rest for two hours, or as much longer as possible. It is then allowed to run from the acid pan into an iron tank containing a weak lye of caustic soda, and boiled up for a quarter of an hour, and after settling for at least two hours, it is sent into the jacketed charcoal tank. In this process of acidification there is plenteous evolution of sulphurous acid, the destroyed hydrocarbons are not left, as in the low-heat treatment, as a sulpho-acid tar, but decomposition has been carried to such an extent that only insoluble charcoal is left mixed with the paraffin.

The wax-filtering apparatus is well known. A skeleton cylinder of tinned iron is covered round its sides with closely perforated tinned iron sheet (zinc will not do). There must be a band at top and bottom, not perforated. It is made with a dished bottom. A belt of flannel is tied round it, and over that a piece of web filtering paper. The paper must come over the landings, and be fixed at the edges by strong paste made with flour and skimmed milk; and to make all secure, a piece of strong twine may be tied tightly round at top and bottom. This is set down in a steam-cased vessel, similar in shape, but 6 inches larger in diameter. A

short pipe, on bottom of inside filter, is fitted into a tapered hole through the outer vessel, and a cross-bar above keeps the two in close contact. The wax flowing in between the inner and outer vessel percolates through the paper into the inside vessel. When the valve governing this is opened, the wax is carried away by a pipe or gutter either for blocking or to the candle house.

The sediment containing at least 70 per cent. of wax has always been found difficult to deal with. By boiling up with water and free steam so as to waterlog the charcoal, much of the paraffin will be liberated, and floating on the water can be decanted off. The residue still contains a large percentage of paraffin, and to recover this, the material is occasionally washed with blue oil, the product being sent to scale. In the latter case the char residue is difficult to get rid of, as those who make extraction a business cannot make much profit off it, the quantity and the quality of the contained wax being much reduced. There are some good extractors in the market, but the material is not suited for treatment by percolation, it must be treated by naphtha washings and stirring, followed by settling and decantation, the naphtha being recovered both from the wax and the char by distillation.

The hydraulic press shown in Figs. 86–88 will, for the pressing of the sweatings, suit better if two-thirds of the plates are removed and replaced by flakes of wicker. Ten hundredweight of pressure on the area of the ram is sufficient for this soft material.

Several materials have been substituted for charcoal in the above process. In one instance, the addition of about 12 per cent. of powdered fullers' earth, at a temperature of 230° F. (110° C.) is recommended. The mixture is well agitated, then left to settle, and the clear paraffin is run off.

Fig. 91A shows an improved apparatus made by S. H. Johnson and Co., of Stratford, for the carrying out of this process, and it may be used also for the treatment of oils and fats with fullers' earth, animal charcoal, or any substance of like properties, such as the silicates of magnesia and of other

bases (Smith and Field's process). As the figure shows, the mixing pans, which may be of any size, have steam-cased bottoms, and are fitted with steam-driven agitators. Fixed to the presses are also steam-driven force pumps, for sending

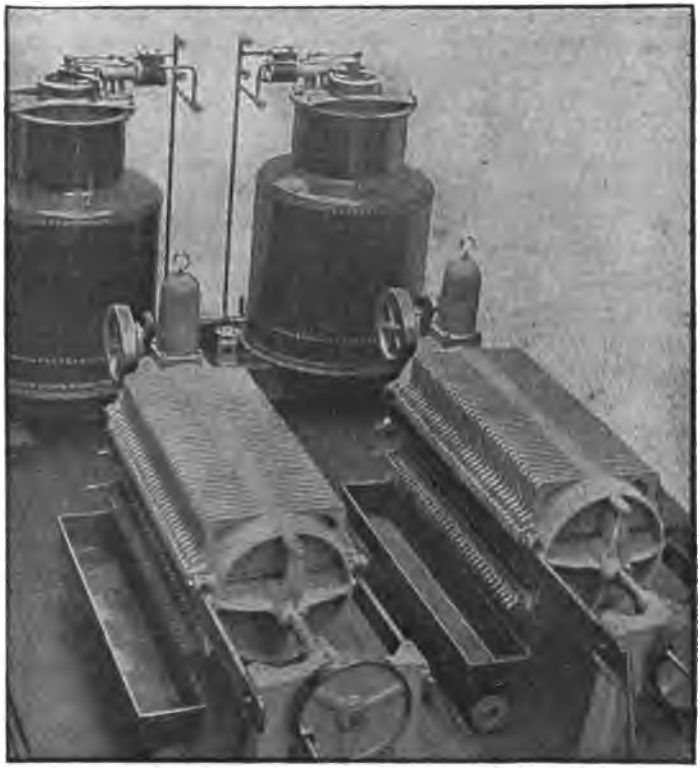


FIG. 91A.

the treated material into the presses. The clay may in a great measure be freed from the wax by forcing hot steam through it after the bulk of the wax has passed through. The same kind of filter press will be found most useful for the recovery of the weak paraffin from the expressed oil from

residues, (8) p. 342, after treatment with acid and alkali, distillation, and cooling.

There has been a very simple press of like construction, with disconnected chambers, made for charcoal treatment of paraffin, the pressure being merely got from a short column of the material, and the filtering medium being discs of paper supported on grooved plates. It is said to be efficient and very speedy. The presses figured are fed through end, and chambers are connected by central holes in supporting plates.

For stock-taking purposes, all the tanks used in the process should be numbered, and the weight of their contents for every inch of depth tabulated, so that by means of a dipping rod, after the water has been run off, the weight of wax may be quickly ascertained. Forty-seven pounds per cubic foot, or 48 cubic feet per ton, will be found an average weight for wax in this condition.

Pure paraffin is sometimes used alone for candle-making; but it is generally mixed with proportions of hard stearic acid varying from 5 to 50 per cent. The refined paraffin causes the candle to burn with a flame of great power, while the addition of the stearin renders it less liable to bend under the influence of a warm atmosphere, or to give off smoke during combustion.

Chemically speaking, the paraffins are true hydrocarbons, and the solid paraffins are the higher terms of the series of which marsh-gas, or methane, CH_4, is the lowest; their general formula is C_nH_{2n+2}. The second term, ethane, C_2H_6, is noteworthy as being the parent of ordinary alcohol, ether, and acetic acid. The sixteenth term, hexdecane, $C_{16}H_{34}$, is solid at 70° F. (21° C.), and the melting-points of the series rise according to their complexity of composition.

The amount of solid paraffin produced in this country, together with that imported, is 50,000 tons per annum.

Ozokerit, or Earth-wax.—Although the viscid forms of native bitumen are very numerous, and widely distributed over the world, it is somewhat remarkable that a solid,

high-melting-point paraffin is only known to occur in Galicia, where miners' candles have long been made from it. The colour of the mineral varies from brown to greenish and yellow tints; its fracture is resinous. It contains about 85 per cent. of carbon, and 15 per cent. of hydrogen, and appears to consist of a group of hard, solid hydrocarbons, whose melting-points range from 140°–176° F. (60°–80° C.). Dr. Letheby says that the illuminating power of ozokerit exceeds that of the best paraffins, and is therefore far beyond those of spermaceti, wax, and stearin. The following table shows the number of grains of the various substances enumerated, required to give the light of 1000 gr. of the best spermaceti candles:—

Ozokerit	754
Paraffin	798–891
Spermaceti	1000
Beeswax	1150
Stearin	1200

The leading properties of ozokerit are:—(1) That it has a very high melting-point, and does not bend or soften so readily in a warm atmosphere; (2) that it has great illuminating power; (3) that it burns with a dry "cup," and is not so liable to "gutter" as ordinary transparent candles; (4) that it is entirely free from smell, is not at all greasy to the touch, and has an appearance not unlike that of the finest bleached beeswax.

To obtain commercial products from the mineral, two processes are employed. One consists in dissolving it in some spirit, filtering the solution through charcoal, and distilling off the spirit from the filtrate. In the second, and most usual, the ozokerit is first heated with fuming sulphuric acid, and then carefully distilled with superheated steam in an apparatus similar to that which will be described in the next chapter, for the distillation of the fatty acids of tallow and palm oil. By Field and Siemssen's patent, the crude wax is heated and distilled *direct* with superheated steam, without previous acidification.

The process of purification by acidification with strong sulphuric acid gives ceresin, a substance much resembling beeswax in consistency and fracture. By this method the *whole* of the mineral is converted into a homogeneous yellow substance, without much loss, except that due to filtration, and a certain amount of charred products. This substance (ceresin) is, however, useless in candles, as it smokes persistently. By Field and Siemssen's patent, the ozokerit is melted, and introduced directly into the still, whence it is distilled by fire heat and superheated steam. This process yields about 50 per cent. of hard white material (140° M.P.), 35 per cent. of black paraffin, 170° M.P. (employed in insulating materials for electrical purposes), and 15 per cent. of oil and soft grease (ozokerine, a substitute for vaseline).

When thus purified the ozokerit resembles fine beeswax in colour, but is more translucent than wax, though less so than paraffin. The hardness and high melting-point [142° F. (61° C.)] of the candles made from this source give rise to a drawback common to wax candles, viz. the smouldering of the wick on extinction. The immediate cause of this is the fact that the cup of the candle dries and solidifies as soon as the flame is blown out, so that there is no liquid matter left to extinguish the spark. This difficulty is now overcome by a special contrivance of the wick.

Pielsticker refines crude ozokerit by agitation first with sulphuric acid, and after treatment with barium carbonate and caustic soda.

The writer desires to express his thanks to Mr. James Bryson, of Pumpherston Oil Co., Linlithgowshire, for drawing of his arrangement of Henderson's sweating closets.

CHAPTER XIV.

PROCESSES FOR THE CONVERSION OF NEUTRAL FATS INTO FATTY ACIDS—THE MANUFACTURE OF COMMERCIAL "STEARIN."

IN the first chapter of this book some account was given of the theory of the constitution of fats, oils, &c., and of the masterly researches of Chevreul into their real nature; in the present one, an acquaintance on the part of the reader with what is there stated will be tacitly assumed. From a keen eye like Chevreul's it was impossible that the intrinsic value of his discoveries could long remain concealed, and in 1825, in conjunction with Gay-Lussac, he started a factory for the manufacture of stearic acid. Chemical knowledge, however, is seldom united in the same person with technical —or rather commercial—success, and the enterprise failed. It was reserved for de Milly to reap the harvest which the great chemist had sown, and in 1832 he manufactured near the Barrière de l'Étoile some excellent stearin (i. e. stearic acid) candles which were called by that name.

One of Chevreul's discoveries was that the removal of glycerin from neutral fats, i. e. their conversion into fatty acids, enormously increased their hardness and illuminating power, so that candles made from the mixture of stearic and oleic acids, resulting from the removal of glycerin from tallow, were less greasy, and gave much more light, than candles made from the same tallow untreated, though they had not so nice a colour. The next step was the separation of the harder from the softer portions of the fatty acids, and it was found that when this was effected by pressure the oleic acid, in flowing away, carried with it in solution the whole of the

colouring matter of the mass, leaving the crude stearic acid tolerably white. To make it absolutely so, little else was found necessary than repeated pressings at various temperatures, the series of operations, after the removal of the glycerin, being purely mechanical. In carrying out this on a manufacturing scale, the expensive alkalies soda and potash were soon replaced by lime, and the preparation of stearic acid by this process is now conducted as follows :—The tallow to be saponified is placed in a large, slightly conical, wooden tun, which is made of oak or cedar, and is tightly bound with iron hoops. Steam is introduced by means of a spiral copper tube, laid on the bottom, and perforated with numerous small holes. An upright wooden shaft, carrying wooden arms fitted with teeth, is fixed in the centre of the tun, and revolves during the process. The tuns are arranged in rows in a large room, two being required for the completion of each batch.

In this tun, the tallow is mixed with good slaked lime, made into a thin cream with water. At first, and even for several years, a very large quantity of lime was used, as much as 16 lb. per 100 lb. of tallow, but this was gradually lessened. After tightly closing the tun, steam is introduced from a pipe below, and the contents are boiled for 3 hours. During the boiling the mixture is kept constantly agitated by means of a wooden shaft bearing some horizontal arms, worked by steam power. The action of lime upon the constituents of tallow decomposes them, glycerin being set at liberty, while calcium stearate and oleate are formed. The formation of these salts, which, when mixed together, constitute an insoluble soap, technically called "rock," greatly facilitates the subsequent separation of the solid and liquid constituents of the tallow. To ascertain when the operation is complete, a small portion of the boiling mixture is drawn out in a ladle, and cooled. When cold, the sample should appear perfectly cool and solid, and should be very brittle, powdering finely in a mortar. When the operation is complete, the steam is shut off and the agitator is stopped, the

whole contents standing until cool, and the fatty matters and lime form a solid mass at the bottom. They are then dug out and removed to another tun, similar in all respects to the last. Here they are treated with 4 parts of strong sulphuric acid for every 3 parts of lime previously added, and are then heated and agitated in the same manner as before. During the operation, the lime salts are decomposed by the acid, calcium sulphate falling to the bottom, and the fatty acids rising in a thick layer to the surface. Again, the whole is permitted to stand; when cool, the fat is skimmed off and placed in a third wooden vessel, where it is well washed with water and by steam blown into it. The washed fatty acids are next heated to the melting-point, and run into dishes or troughs made of tin; these are placed in a room, the temperature of which is kept at 68°–86° F. (20°–30° C.), and left for 2 or 3 days, or until the contents have assumed a granular or crystalline structure, when they are removed from the dishes, and placed in canvas or woollen bags, or between large square sheets of canvas, and are carefully deposited between the plates of a powerful hydraulic press. Pressure is exerted gently at first, and is gradually increased until the flow of the liquid oleic acid ceases. The press is then unlocked, and the hard thin cakes of crude stearic acid are thrown into another similar wooden tun. Here they are melted down by blowing in steam, which is continued for some hours. After settling, the fatty matters are drawn off into tin dishes, and placed aside to cool. The temperature of the room in which the cooling is conducted should be slightly higher than the previous one, or about 86° F. (30° C.). The dishes should remain here until the contents assume a crystalline structure, when they may be emptied. The blocks are then cut up into lumps, and ground to a mealy powder by means of a rasping machine, worked usually by steam. This powder is gathered into bags, made either of hair or of wool, or both, and is then submitted to a second pressure in another hydraulic press, differing from the former one by having a heating apparatus attached; the plates

should also be heated before the press is used. The temperature and pressure vary with the material operated on, but with good tallow, a pressure of 6 tons per sq. in. at a temperature of 120° F. (49° C.) is not uncommon. The necessity for heat in this second pressure is due to the extreme difficulty experienced in eliminating the last portions of oily matter from the fat. When the full pressure is being exerted, the press is left for about 15 minutes before being unlocked. The cakes thus obtained are cleaned with a knife, the parings being added to the next batch. They are again melted by steam, a little wax being sometimes added, in order to destroy the crystalline texture of the stearic acid, which renders it unfit for use in candle-making.

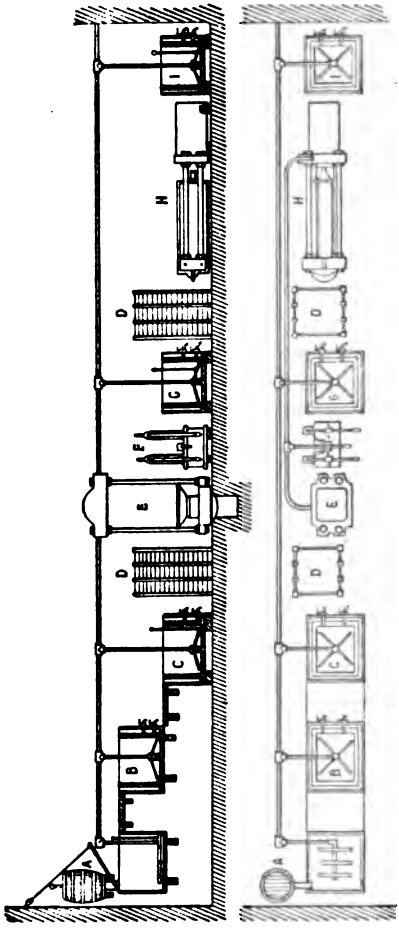


FIG. 92.—APPARATUS FOR PRODUCTION OF STEARIC ACID BY LIME SAPONIFICATION AND PRESSURE.

The arrangement of the apparatus is shown in Fig. 92: A, tub from which milk of lime is supplied; B, lead-lined vats with steampipe, for boiling fat and lime; C, ditto to which "rock" is transferred, and boiled

with sulphuric acid; D, rack holding pans for caking mixed acids; E, cold press; F, hydraulic pump; H, hot press; I, vat for melting the refined stearin for casting into blocks.

This finishes the process, and the stearic acid, commercially called "stearin," is melted into blocks ready for use. The oleic acid as it runs from the presses still contains much stearin, which is separated from it by filtration, exposure to artificial cold, &c. As the relative prices of stearin and olein usually range from about 3 to 2, attention to these precautions is most necessary.

The disadvantage of this process is that so much sulphuric acid is required to decompose the lime soap, thereby injuring the colour of the resulting fatty acids. The process of decomposition with excess of lime, as above described, is now practised to a limited extent. It was soon found that if the saponification were conducted in closed vessels, under a steam pressure of 3 or 4 atmospheres, the amount of lime might be reduced to about 3 or 4 per cent. upon the tallow, thus lowering the cost, and improving the colour, of the product. This modification is still very largely worked, especially in America. Subsequently it was discovered by Tilghmann, who patented the process in 1854, that if sufficiently high temperature and pressure were employed, the lime might be dispensed with altogether, and that the resolution of the fat into fatty acids and glycerin might be effected by steam alone. This process, known as the "autoclave," has been largely worked, both in Europe and America; but in consequence of numerous accidents, arising from the explosion of improperly constructed vessels, it is usual to decompose tallow at a lower pressure, with the aid of 2 or 3 per cent. of lime, the subsequent operations of crystallisation of the fatty acids, hot and cold pressing, &c., remaining the same. As at present conducted by the large London candle-makers, about 1 ton of fat, usually mixed tallow and palm oil, is heated with 2 per cent. of lime, and $\frac{1}{3}$ the fat-volume of water, in an upright Papin's digester, at a pressure of 8 atmospheres (about 110 lb.) for 4 hours. The whole is

then blown-out into a tank, and the "sweet-water" is run off. The subsequent processes are detailed above. Another recent modification of this process consists in replacing the 2 per cent. of lime in the above operation by a fraction of 1 per cent. of zinc oxide; this is fully described in the chapter on Glycerin, and will probably prove of more value to the soapmaker than to the candle manufacturer.

The subject has recently been discussed by an anonymous author,* who shows that the later stages of the process are very tardy, indicating that after 3 hours' operation but little extra result is attained. He also mentions that saponification by MgO is less complete than with CaO.

The next advance was the discovery that when neutral fats are exposed to a very high temperature, 572° F. (300° C.), or above, in presence of superheated steam, they are decomposed, and the fatty acids are volatilised; and that when these vapours are condensed, the fatty acids are almost white: that, in fact, fatty acids may be distilled, almost unchanged, in an atmosphere of superheated steam. It was impossible however, to conduct this process on a large scale, in consequence of the simultaneous production of acrolein, a vapour resulting from the decomposition of glycerin, and possessing intensely irritating properties; but in 1841 it was discovered that if neutral fats were treated first with concentrated sulphuric acid, and then boiled with water, they might be distilled without any such inconvenience, and the problem was thus solved by Dubrunfaut. In 1842 and 1843 Jones and Wilson, under the name of Price & Co., took out two patents for the combined treatment of fatty bodies by sulphuric acid and water successively, and their subsequent distillation by the aid of superheated steam. From that time to the present this process has been worked, in its various modifications, on a most extended scale, especially in England. It gives a much larger quantity of material, of good colour for candle-making, from a given weight of fat, than any other known process. The candles are not so hard,

* Journ. Soc. Chem. Ind., xii. 163.

nor quite so white, as the Continental *bougies* of stearic acid; but while tallow, treated by the saponification process, yields only about ½ its weight of candle material, when acidified and distilled the yield of stearin is greater by about 10–15 per cent.; its quality, however, is slightly inferior.

The most perfected form of apparatus now used in the distillation process, as made by Merryweather & Sons, of London, is shown in Fig. 93. The process is conducted as follows:—The fat is melted from the casks in which it is stored by means of a steam jet inserted in the bunghole, and runs into the underground wooden tank A, where it is left for some hours to settle the condensed water out of it. Hence it is pumped, by means of the gun-metal lift- and force-pump C, into a series of

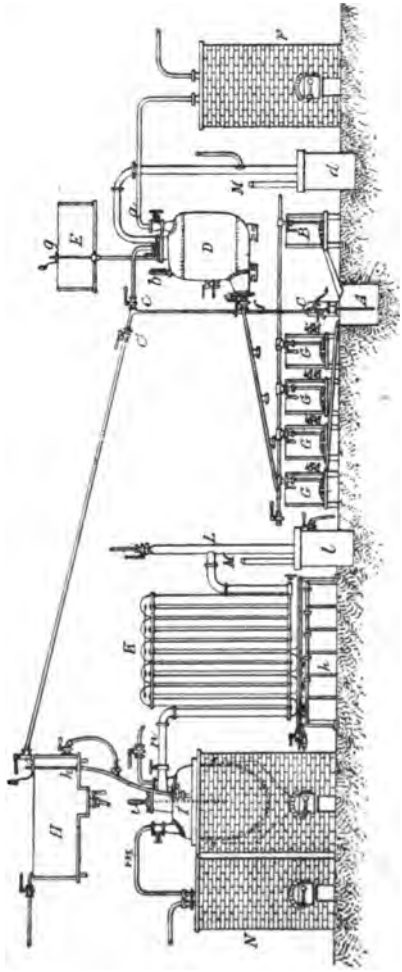


FIG. 93.—APPARATUS FOR DISTILLATION OF FATTY ACIDS.

lead-lined collecting tanks B, fitted with steam coils, by which the material is boiled before being passed through the tap *c* to the vessel D. This latter, which is known as the "acidifier," is made of stout copper, supported either on wrought-iron girders or on brickwork. It is fitted with a valved pipe *a*, for the admission of superheated steam; a copper pipe with water-shower pipe *d*, for condensing the vapours generated by the acidifying process; a thermometer *b*, for guidance as to temperature; also a gun-metal cover *e*, at the lower side, for cleaning out, and to which is affixed a tap *f*, for drawing off the acidified materials. On admission to D, the fats are heated for a certain time by the introduction of superheated steam, at a temperature of about 350° F. (176° C.), from the superheater F, constructed from the special design of Ed. Field, C.E.

Sulphuric acid in the proportion of 3–6 lb. per cwt. of fat is next supplied to the acidifier from the tank E by opening the plug *g*. When the acidification is complete, the material is left to stand for about 6 hours, and is then discharged into a series of lead-lined, open washing-vats G, provided with copper steam coils, and containing water and a little sulphuric acid. Here it is boiled with free steam for another 2 hours, and is left for about 24 hours to settle; it is then drawn off into the tank A, and pumped through the tap *c'* into a large, open, lead-lined tank H, placed at a sufficient elevation. This tank is fitted inside with a coil, which is charged with steam, to keep the contents in a liquid state. By means of the valve *h*, about 5 tons of the material is run into the still I, consisting of an iron body and copper dome; it is fitted with a thermometer *i*, and the necessary taps of copper or gun-metal. The contents of the still are heated by fire to a temperature of about 240° F. (116° C.); superheated steam, at about 560° F. (294° C.) is then admitted by the pipe *m* from the superheater N, and the process of distillation commences. The temperature must be regulated according to the quality of the material operated upon. The vapours pass over by the pipes *n* to the refrigerator K, which consists of a series of vertical copper pipes, connected at top

and bottom by gun-metal bends. These pipes are mounted on iron frames, over a set of 6 circular iron tanks *k*, into which they can be emptied. The tanks are furnished with pipes for the admission of steam, and with spiral copper cooling-coils, through which cold water may be passed. The "essence-tank" *l* is fitted with an improved shower-pipe L, which prevents any vapour passing away uncondensed. The pipe M conveys vapours to be burnt in the flue.

The fatty acids are collected in pails from the mouths or outlets of the copper coils, and may be used direct for certain low qualities of candles. It is usual, however, to crystallise and press them as already described. The residue remaining in the still is transferred to another distillation vessel of iron, in which it is subjected to great heat; a further instalment of fatty acids is thus obtained, which is pressed as above. The ultimate residue in the still is known as "pitch," and is recognised as a commercial article of many uses, and will in all probability soon be recognised as an efficient substitute for "black-japan," for coating iron. The approximate cost of the plant required for distilling tallow or palm oil according to the above process, exclusive of steam boiler, may be stated at 1700*l.* to 3150*l.*, according to whether 1 ton or 3 tons are to be distilled at a time. As the result of applying the combined processes of acidification and distillation to tallow, 100 parts will yield about 60 per cent. of stearin, 28 of olein, and about 2 of pitch; the exact yield of stearin depending of course, upon its setting-point, and the quality of the tallow from which it has been derived. The acidification process, when followed by distillation, is often employed as a means of purification of fatty acids obtained by any of the other processes of decomposition.

Palm oil is now used in enormous quantities for the production of palmitic acid at the works of Price's Patent Candle Company, as well as by other candle manufacturers in Great Britain, about 25,000 tons being annually consumed. In many Continental countries, a prohibitive duty prevents its employment. The plant and the *modus operandi* scarcely

differ from those last described. The distilled mixture of palmitic and oleic acids is cut into shreds, by means of a revolving knife, and the shreds are wrapped in canvas or woollen cloths, spread in even layers between mats of cocoanut fibre, and submitted first to the cold press and afterwards to the hot press, at a temperature of 85°–90° F. (29°–32° C.). The pressed cakes of fatty acids are pared, and then melted again by steam, in large, wooden, iron-bound vessels, containing water and sulphuric acid. The whole is boiled for a time and is then allowed to stand, after which the acidulated water is drawn off. The melted fatty acids are repeatedly washed with hot water, and then run into moulds; when cold, they are quite pure, and ready for manufacture into candles, after admixture with stearic acid obtained from other fats.*

Of late years, a quantity of inferior stearin (using the word in its commercial sense) has been prepared from what is technically known as "recovered grease." In spinning wool, it is necessary to lubricate it with a certain amount of oil, which afterwards has to be washed out. From these and other processes in woollen mills, large quantities of wash-waters are run to waste, containing soap in solution, and oil in suspension. In many factories, and especially in those localities where it is forbidden to pollute the rivers, these wash-waters are run into tanks, where enough mineral acid is added, to give them, after thorough agitation, a faintly acid reaction. After some hours, the contents of the tank divide themselves into (1) a greasy dirty scum, (2) clear water, (3) greasy mud on the bottom. The clear water is run away, and the residue, after draining, is packed in canvas bags and placed in hydraulic presses, which are slightly heated during the operation. Water and crude fatty acids are expressed, and separate from each other by subsidence; the fatty acids are then pressed and distilled as described

* In the Journal of the Society of Arts for 1852 is an excellent lecture upon the history of the stearin industry, by G. F. Wilson, of Price's Patent Candle Company, who should perhaps rank next to Chevreul for his share in developing it.

above, the yellow hard portions being used for low class candles, and the oil used over again in the mill.

It will be observed that 3 processes for the decomposition of neutral fats have now been described—viz. (1) By saponification with a strong alkali, at a temperature but little above 212° F. (100° C.); (2) By the use of water, with or without a very small quantity of lime, at very great steam pressure, and a correspondingly high temperature; (3) By treatment with strong sulphuric acid and water in successive portions, and subsequent distillation at normal atmospheric pressure, but at a dangerously high temperature—above 572° F. (300° C.). It was reserved for a physician at the Danish Court, the late Dr. J. C. A. Bock, to demonstrate the important fact that, by properly conducting the operation, water alone might be made to decompose tallow into fatty acids and glycerin, and that by the use of water and sulphuric acid combined, fatty acids might be prepared from tallow in open lead-lined tanks furnished with steam coils,—without any of the complicated and dangerous apparatus required by the "autoclave" or the "distillation" processes, without any lime or other alkali, and with a much less expenditure of acid than was required by any other process. Unlike many inventors, he was able to carry out his ideas into actual practice, and in the International Exhibition held in London in 1862, were shown some beautifully white and hard stearic acid candles, which had been prepared by this process in the manufactory of O. F. Asp, Prindsessegade, Copenhagen. Since then, the process has been constantly at work; it has also been adopted in several other Continental candle factories, and among other places, in New Zealand. The simple character of the "plant" required renders it peculiarly valuable for countries distant from great manufacturing centres. Considered from a theoretical point of view, it is perhaps, the most ingenious and the most strictly scientific of all the methods for decomposing neutral fats.

Dr. Bock pointed out that tallow is composed of exceedingly minute globules of fat, surrounded by membranous

envelopes, composed, probably, of albumen; and that until these enveloping walls are destroyed, no reagent can act upon the fat within. In ordinary saponification, the albuminous envelopes are dissolved by the caustic alkali; in acidification, they are burned and charred by the strong sulphuric acid, the quantity of which may be so adjusted as not to burn and discolour the tallow itself, which, after pouring out from the destroyed envelopes, is in a state to be readily decomposed by water at 212° F. (100° C.) Dr. Bock's process was described by him in an article in Dingler's 'Polytechnisches Journal,' for May 1873, of which the following is a synopsis.

By the lime saponification plan, the albumen contained in the fat is dissolved, lime-soap is formed, and the bulk of the glycerin having been set free from its combination with the fatty acids, may be separated in the most favourable condition for after treatment. By the acidification process (see p. 356) the treatment with strong acid relieves the fat, chars the tissue, decomposes most of the fat, and destroys the greater portion of the glycerin, leaving the residue in the worst condition for purification. Acidification, rationally conducted, is only a preliminary operation, intended to break up, corrode, or carbonise, the albumeniferous matters. But the conduct of the operation was long based on the erroneous belief that a double acid, sulpho-stearic, was formed. With due care, only the envelopes of the cells are blackened, and these are soluble neither in fat nor in fatty acids. The production of a real black solution is only an evidence that a certain part of the fat has been burned, which should be avoided under all circumstances. There is no doubt that the operation has generally been carried to excess, in the matters of duration, height of temperature, or strength of acid. By proper acidification, the neutral fat is only unclothed, as it were, and freed from the cells, or at any rate, the latter are so ruptured as to allow of the easy exit of the fat. This latter is then in a condition to be decomposed, an operation accomplished in much shorter time by the chemical equivalent of acid—

4–4½ per cent.—and the necessary water. After letting out the glycerin waters, the fatty acids appear more or less black. They may now be distilled. Their melting-point varies from 120° to 134° F. (49°–57° C.).

The real value of Dr. Bock's method consists in dispensing with distillation. The object of this operation is the removal of the back colour, or rather of the black-coloured matters, by superheated steam. These black matters are the partially carbonised albumen cells, which swim about in the fatty acids because the sp. gr. of the two bodies is about the same. This difficulty is overcome by oxidising the mass, by which the sp. gr. of the cells is raised from 0·9 to 1·3. They are thus precipitated, and the fatty matters can be washed off. The subsequent cold and hot pressings are the same as with ordinary methods.

From several years' experience at Asp's works, the following results have been deduced :—Tallow yields, by complete decomposition, 95 per cent. of fatty acids, which lose 2 per cent. by oxidation and washing. The glycerin obtained from tallow equals 6⅔ per cent. at 1·190 sp. gr., and is quite free from all organic acids. The oleic acid resembles that produced by the lime saponification process; but it is much richer in solid acid. The stearic acid is also like that produced by the lime saponification method; but it is much harder, and its melting-point is 136°–140° F. (58°–60° C.). It amounts to 55–60 per cent. of the tallow employed.

The plan is free from danger, as the steam is only used in open vessels. The plant is much cheaper, as nothing special is required. The labour also is much reduced, as the operation is completed in one vessel. It is as applicable to vegetable as to animal fats.

The process indicated above has now been for many years in daily operation on a manufacturing scale in Copenhagen, and, in the hands of the inventor and his son, Alexander Bock, has been greatly improved and simplified since its first introduction. At that time, there were 5 stages in the process, viz. :—(1) Acidification, to remove the membranous

cellular tissue from the tallow; (2) Decomposition, by acidulated water, into dark fatty acids and glycerin; (3) Oxidation, to increase the sp. gr. of the dark membranous matters, so that they might separate themselves from the fatty acids; (4) Repeated washings with water; (5) Pressing, both cold and hot.

The greatest improvement is due to the discovery that the dark membranous matters may be oxidised while the fat is still neutral. The acidification, oxidation, and decomposition are all now conducted, in rapid succession, in one and the same wooden tank, after which, one or two washings in another tank render the fatty acids fit for the press.

Another point of great practical importance, which has been developed in the working out of this process, is the increased hardness of the stearic acid produced by it, arising from the solidification of some of the oleic acid in the tallow, by the prolonged action of sulphuric acid upon it. This reaction has lately been pointed out as a novelty, by Bornemann and Kraut; but it was suspected some years ago, and soon afterwards was definitely proved, by Bock. He observed that the fatty acids became harder and harder, so that cold pressure had no effect upon them; by the adoption of hot pressing, he produced white stearic acid, and an exceedingly brown oil. This latter, when distilled, saved the cost of distillation, by its yield of solid matters. It is claimed, therefore, for the Bock process, that stearic acid can be made of better quality and in larger quantity from a given weight of tallow, than by any other process at present known.

Since the above account of Bock's process was written, ten years ago, the conviction has been forced on many that it has been overpraised. To the present writer's knowledge, and also from information obtained from more than one trustworthy source, this process was tried by the largest firm in Australia, and others, and after a time abandoned, not because it was impracticable in working, or that a good quality of stearic acid was not got, but that, on account of the small proportion of solid candle-making material obtained,

it would not pay. That this is a pretty general verdict is evident from the fact that firms in the first rank have not adopted it, and still continue to order and use autoclaves and other machinery of the most expensive description.

The process wrought at present by those large and wealthy firms who can afford the outlay is a compound one. The several steps are—(1) Decomposition in autoclave by superheated steam in presence of a small percentage of caustic base, such as lime, magnesia, or baryta (see p. 353). This decomposes the fat, while in a great measure the glycerin is conserved. (2) Acidification in a copper vessel (see p. 355, Fig. 92, D) with small percentage of sulphuric of 1·82–1·84 sp. gr., the quantity being varied according to the character of the material used, and its temperature. It is here that, by the conversion of oleic acid into elaidic or some such like solid acid, the economy of the method over others lies. The heat to which it is raised varies from 115°–121° C. (240°–250° F.) to in some cases 166° C. (330° F.). (3) Distillation by aid of superheated steam (see p. 355). (4) Cold and hot pressing, as described on pp. 351–2.

For further information regarding this subject the reader is referred to Groves and Thorp, Chem. Tech., ii., "Stearine," to the author of which article, Mr. John McArthur, the writer is much indebted for his kindness in overlooking and suggesting alterations in the proofs of Chaps. XIV. and XVI.

Many manufacturers, and notably small ones who cannot afford the expensive machinery, begin their process at the second or acidification stage, especially with fats containing a small proportion of glycerin (see pp. 36–37).

Müller-Jacobs mixes oils at 140° F. (60° C.) with 30–40 per cent. of sulphuric acid at 164°–165° Tw. (65° B.). When the mixture has a temperature of 95° F. (35° C.), it is mixed with double the volume of cold water under constant stirring. The sulpho-fatty acid produced, after boiling for 24 hours, is separated and boiled continuously in several times its volume of water, being thereby converted into a solid fatty acid,

resembling stearic acid, melting at 159° F. (70½° C.) and into liquid oxyoleic acid. From the separated fatty acids soluble in alcohol, the solid constituents are allowed to crystallise out by standing 2–3 days in the cold, after which the liquid contituents (say 40 per cent.) are separated by centrifugals or presses, and the cake is washed and distilled.

Recently Stein, Bergé and De Roubaix have patented the application of a solution of sulphurous acid or an alkaline bisulphite as the hydrolytic agent. Some 2½–3 per cent. of the solution is introduced upon the fat in a vessel capable of withstanding great pressure, and the temperature is raised to 338°–356° F. (170°–180° C.), producing a pressure of about 18 atmospheres. Care must be taken that the temperature does not exceed 392° F. (200° C.). The operation is completely performed in about 9 hours.

Knab's apparatus for conducting continuous distillation by superheated steam presents some peculiar features. The fatty acids are run into the distillation vessel by means of a supply funnel at intervals regulated by the rising and falling of a float valve. Superheated steam is admitted by a pipe furnished with a cock and safety valve, and passes in small streams through the molten fatty acids from a horizontal coil lying at the bottom of the vessel. The vapours pass off by a neck to a series of condensers, from which the distillates are separately drawn. A blow-off pipe permits the discharge of residual pitch at intervals, during which operation the supply of fatty matters is temporarily suspended. The vessel is heated by immersion in an outer shell containing molten lead.

Marix attaches an air-pump to the still, so that the distillation is effected at reduced pressure and at temperatures not exceeding 482°–491° F. (250°–255° C.).

Lewkowitsch adopts similar measures, thus diminishing the pressure by 10 to 13 lb., and lowering the temperature from about 600° F. (316° C.) to 460° F. (238° C.).

The temperature of distillation influences the colour of the distilled fatty acids: at 464° F. (240° C.), they are white;

at 500° F. (260° C.), just coloured; at 554° F. (290° C.), more coloured; above 572° F. (300° C.), yellow to brown.

The consumption of steam for every 1 part of fatty acids distilled at different temperatures is given by Schädler thus: 7 parts at 392°–446° F. (200°–230° C.); 3–4 parts at 446°–500° F. (230°–260° C.); 2 parts at 554° F. (290° C.); 1 part at 617°–673° F. (325°–356° C.).

Conversion of Oleic into Palmitic Acid.—Whichever of the 4 processes (lime saponification, autoclave, distillation, or Bock's) is employed, a large proportion of oleic acid is unavoidably produced. The quantity of it, per ton of neutral fat, varies in inverse proportion to the hardness of the original fat, or of the material manufactured from that fat. For many years, it was difficult, at any rate in England, where soft soaps are much less used than on the Continent, to find a suitable outlet for this oleic acid: it could not be used as a lubricant, owing to its acid reaction upon metals; when saponified by the ordinary methods, it produced a very soft and very brown soap, slow of sale. It was discovered, however, that when saponified with soda-lye of very high specific gravity, a hard soap could be made from it, containing a very large percentage of fatty acids, and good for ordinary cleansing purposes, but its smell was considered objectionable. It was, nevertheless, found to possess certain special advantages for many manufacturing purposes, especially in the woollen industry, and accordingly it finds a ready sale under the name of "pure oil" soap (pp. 148, 254). A few years ago, moreover, Radisson, of Lyons, taking advantage of a laboratory reaction of oleic acid which had long been known, developed a method of converting it into palmitic acid, and, by dint of great perseverance, worked out the details of the process on an industrial scale; it is now a commercial success, and has been patented in nearly all countries where candles are manufactured. Whether it is more economical to convert the oleic acid into soap or into palmitic acid, depends upon the relative cost of the two processes, and the current market values of the manufactured products.

The following information relative to this remarkable process, which is extremely interesting, from both scientific and technological points of view, has been kindly supplied to the writer, by the patentee, St. Cyr Radisson, 37 Boulevard Oddo, Marseilles.

In 1841, Warentrapp announced that when oleic acid was heated with a great excess of caustic potash, it was decomposed into palmitic acid, acetic acid, and hydrogen, the acids combining with the potash, the reaction being explained by the following equation:—

Oleic acid.		Potash.		Potassium Palmitate.		Potassium Acetate.		Hydrogen.
$C_{18}H_{34}O_2$	+	$2\,KHO$	=	$C_{16}H_{31}KO_2$	+	$C_2H_3KO_2$	+	H_2

At Marseilles, this has been practically realised, and about 3 tons of oleic acid can be daily converted into palmitic acid, by this process; it is used in Fournier's large candle factory. The system has also been worked in Copenhagen, in conjunction with the Bock process, and elsewhere.

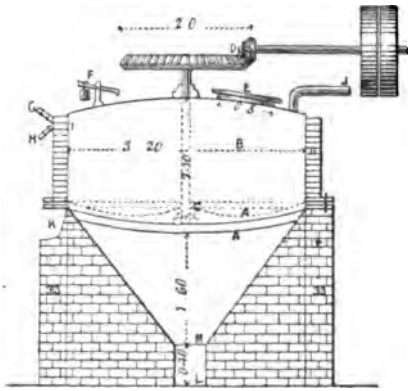


FIG. 94.—CARTOUCHE.

The vessel in which the fusion takes place is technically called a *cartouche*, and is shown in section in Fig. 94. It is a cylindical vessel A B, 10 ft. 6 in. in diameter, and 4 ft. 4 in. in height, the two ends being slightly "dished," and the bottom made of cast iron, while the sides are of wrought iron. The whole is set in brickwork in such a way that the bottom is at least 5 ft. 9 in. above the firebars M, thus giving a large fire-chamber, a very important point in the facility of control of temperature and the maintenance of

a uniform heat. The cartouche contains any convenient mechanical agitator C, worked with gearing D, also a manhole E, safety-valve at F, steam and water cocks G H, exit-pipe J for steam and hydrogen, and a large outlet pipe K, with suitable valve, for removing the fused mass at the end of the operation. In working with caustic potash, the original form of the process, the cartouche is charged with 1650 lb. of oleic acid and 2750 lb. of caustic potash lye at 80° Tw. (41° B.). Heat is gradually applied, and when the disengagement of steam ceases, the manhole E is closed, and the evolved gases pass through J to a coke-tower condenser, and thence to a gasholder. At 554° F. (290° C.) decomposition commences and much hydrogen is evolved. From 572° to 590° F. (300°–310° C.) is the proper temperature for the operation, and at 608° F. (320° C.) the odour of the evolved gas suddenly changes, and the stage of destructive distillation is reached. To arrest this, steam is suddenly distributed through the mass, by the Giffard injector I. Some 36–40 hours are required for one complete operation, including filling and emptying; its progress is judged by taking samples, decomposing them, and determining (by Dalican's method, p. 99) the solidifying point of the fatty acids. When the outlet-pipe K is opened, the potassium palmitate falls into an open tank, where the soap, and a quantity of water sufficient to melt it, are heated together by a steam-jet. After subsidence, the contents of the tank divide themselves into an upper layer of neutral potassium palmitate, and a lower layer of potash lye, usually about 28½° Tw. (18° B.). The neutral palmitate is removed to another vessel, and decomposed with sulphuric acid; the last traces of potassium sulphate are removed by washing with water.

At this stage, the palmitic acid is of a clear chocolate hue, and when cooled, crystallises in large tables; its solidification-point varies between 122° and 127° F. (50°–53° C.), according to the nature of the oleins employed. It can be distilled with great facility in the usual apparatus, and

leaves only 3 per cent. of pitch. After distillation, the palmitic acid is extremely white, and burns with a very clear, smokeless flame. Moulded into candles, it compares very favourably with the best stearic acid; and when mixed with ordinary stearic acid, it "breaks the grain" of the latter (i. e. destroys its tendency to crystallise), and gives it a semi-transparency, very valuable in the eyes of a candle manufacturer.

Instead of decomposing the potassium palmitate by sulphuric acid, it may be boiled with milk of lime, under a pressure of 3 atmospheres, when the result will be a lime soap, floating in caustic potash lye. So much water, however, is necessary for this reaction, that the resulting lye is only 9° Tw. (6° B.), and its concentration to 85° Tw. (43° B.), is so costly, that it is more economical to regenerate the potassium sulphate by Leblanc's process. The potash lye may be completely causticised in the cold at a sp. gr. of 32½° Tw. (20° B.) with 6 hours' brisk agitation, thus economising fuel. The calcium carbonate so obtained is powdery, and can be easily washed by "displacement" (i. e. running water through it to wash out the potash), in a layer 3 ft. thick. The caustic lye is rapidly concentrated to 85° Tw. (43° B.), and stored in tanks, where, on cooling, it deposits the small amounts of potassium sulphate and carbonate which it contains, and the vertical partitions of the tanks become covered with crystals of potassium acetate, arising from the preceding solidification. The perfectly clear lye is then employed in the transformation of fresh portions of oleic acid into palmitic acid. The crystals of potassium acetate are separated by a centrifugal machine, from the lye which hangs about them, and are then taken to a distilling apparatus, where the acetic acid is displaced by sulphuric acid. The crude acetic acid, thus obtained, is purified by a second distillation, and becomes of commercial value. Its quantity should be 2·5 per cent. of the oil solidified.

Oleic acid which is the product of the distillation of fatty bodies, contains small quantities of hydrocarbons analogous

to natural petroleum. These distil over during the conversion of the potassium oleate into palmitate, and are condensed in a tower, furnished with transverse partitions extending alternately nearly across its diameter. A simple rectification makes them pure enough for illuminating oils, and the paraffin which remains in the heavy portions of the oil can be separated by crystallisation in the cold. As a matter of purely scientific interest, it may be mentioned that caprylic alcohol, sebacic acid, caproic acid, and other substances, are formed in very small quantities, simultaneously with the palmitic acid.

All fatty bodies, with the exception of mares' grease, and the fat of "suint," can thus be solidified by the action of caustic potash; but the ultimate products vary, and the palmitic acid is by no means always pure. Different percentages of palmitic acid are obtained in the final result, according to the nature of the fatty bodies employed. Thus, 100 lb. of oleic acid, resulting from tallows decomposed by lime, should yield 91 lb. of palmitic acid fit for candle-making, while 100 lb. oleic acid resulting from distillation processes only yields 87 lb. of white palmitic acid. It may be noted as an interesting fact, that the oleic acid produced by Bock's process is more suitable for conversion into palmitic acid than that produced by either of the other methods. The two processes (Bock's and Radisson's) are worked conjointly with the most satisfactory results. The percentage of palmitic acid from Bock's oleic acid is higher than from any other.

The cost of the preparation of white palmitic acid by this process is estimated at 13*l.* per ton, but can be materially reduced by the substitution of soda for potash. Owing, however, to the comparative infusibility, and want of heat-conducting power, on the part of sodium oleate, it is necessary to add a quantity of solid paraffin, in which the sodium oleate is suspended during the operation; in this way an equilibrium of temperature is speedily distributed throughout the whole mass, and there is no fear of heating the sodium oleate to its

decomposing-point, since that is higher than the temperature at which paraffin distils. When the mass is treated with water, the paraffin forms the topmost layer, and can be completely separated. The loss in manipulation is found not to exceed 1 per cent. of the oleic acid originally taken, and the cost of the conversion is about 7*l.* 10*s.* per ton instead of 13*l.* Radisson suggests as a still further reduction, the use of heavy hydrocarbon oils for paraffin and of the caustic "red liquors" from a Leblanc soda factory, estimating that the cost might thus be reduced to as low as 10 *francs* per 100 *kilos.*, or about 4*l.* 5*s.* per ton.

Radisson claims the following advantages, as accruing to the stearic acid manufacturer from the adoption of his process:—1. Utilisation of the olein, a troublesome bye-product of variable value; 2. The floating capital necessary for the purchase of raw material is diminished by about 30 per cent., the production of stearin being increased by nearly the amount of olein produced; 3. The manufacturer can use low-priced greases, whose value varies in inverse proportion to their richness in olein; 4. The stearin produced by this process is little, if at all, inferior to that produced by any other process.

De Wilde and Reychler have shown that by heating oleic acid with a small proportion of bromine or iodine under pressure, some 70 per cent. of the oleic acid bcomes converted into stearic acid; and Zürer prepares stearic acid from oleic acid by first subjecting to the action of chlorine, and then treating the dichloride of oleic acid so formed with a current of nascent hydrogen, evolved by the action of water heated under pressure upon finely pulverised iron, magnesium, or zinc. These facts are, for the present, of purely scientific interest, as the cost of the processes prohibits their use in practical working.

CHAPTER XV.

THE MANUFACTURE OF CANDLES AND NIGHT-LIGHTS:—Their Value as Illuminants.

A candle consists essentially of 2 parts: (1) the combustible material; and (2) a porous substance through the medium of which combustion takes place. The first portion of the candle, the combustible material, has been fully dealt with in the two preceding chapters, and the second portion, or the wick, the type of which is found in the rushes employed by our forefathers, is usually made of cotton. The chief essential qualities of a wick are good power of absorption, and a capacity for burning freely, evenly, and thoroughly, while producing the least possible proportion of ash. It must necessarily be quite free from inequalities of whatever kind, and should be made of perfectly sound fibres. The forms and kinds of wick differ widely with the quality and composition of the candle; the melting-points, and other characteristics of the hydrocarbons forming the candle, vary to such an extent that, in order to burn to the best advantage—or indeed, in some cases to burn at all—each sort of candle needs to be accommodated with a special wick. One of the greatest secrets of candle-making is to have the wick perfectly suited to the peculiarities of the fatty matters employed; on this score, it is impossible here to do more than indicate the principles involved.

But little variety is to be remarked in the choice of material for making wicks. The original, and not yet obsolete, medium was the common soft rush, *Juncus conglomeratus*, to be found in most moist pastures, and by the sides of streams and ditches. The rushes are in best condition in the

height of summer, but may be gathered on to the autumn. As soon as cut, they are placed in water, otherwise they would dry and shrink, and the peel would not run. They are then stripped of half the peel, the object of which is to expose the pith sufficiently to enable it to conduct the molten fat, while enough of the rigid epidermis remains to afford it support. When duly peeled, they are laid out to bleach and take the dew for some nights, and are afterwards dried in the sun. These rushes are gathered in Lancashire, and abundantly in the Fen Country, and in Ireland. Candle-wicks are ordinarily made of fine cotton yarn; Turkish cotton rovings are said to be the best, but of the cotton employed for this purpose there is certainly a great deal more imported from the United States than from Asia Minor. The wicks of night-lights vary greatly in composition, according to the fancy of the manufacturer. Sometimes little sections of rush are used, as well as very fine cotton yarn; but the majority consist of "inkle," a fine flax yarn.

The manufacture of cotton and flax wicks is now performed almost exclusively by machinery, the threads of fibre being bound together either by twisting or by braiding. For dip candles, the wicks require to be bulky and of loose texture, in order that the melted tallow may rise freely. They are therefore made by twisting, and constitute the simplest form of wick after rushes. The cotton yarn chosen for the purpose must be "oozy" or furry, and the threads must be free from twist. This is placed ready balled in the cutting machine, a simple contrivance already repeatedly illustrated elsewhere. By it, the yarn is doubled in proper lengths around a rod; a knife then descends and severs the yarns, to which a twist is communicated, by means of a rolling apparatus worked by a treadle. The twist is secured by dipping the wicks at once in molten fat.

Twisted wicks have a great drawback, inasmuch as they are only very partially consumed in the flame, and thus necessitate the troublesome operation of snuffing. The first

attempt to remedy this evil was made by Price's Patent Candle Company, on the occasion of the Queen's marriage. The candles were made self-snuffing, by means of plaiting the wick, and "gimping" strings of wire, or other fibrous material, into the plaits, with the object of bending the wick outwards, so that the end of it should reach the oxidising part of the flame, and thus be destroyed. Palmer in his "metallic wick candles" introduced a fine thread impregnated with impalpably powdered bismuth, into the body of the wick, which consisted of parallel fibres bound together by one wound round them. The bismuth fuses into a little ball, the weight of which draws the wick laterally out of the flame into contact with the air, having performed which duty, the bead oxidises and disappears, by volatilisation or otherwise. A simplification of this plan consisted in plaiting the wick with strands of unequal length, by which the same result was attained. In these cases, the wick was round. At the present day, plaited wicks are made flat, by which means they acquire a natural inclination to bend. For all kinds of moulded candles, plaited, or in technical language, "braided," wicks are used, the old-fashioned twisted wick being reserved for "dips."

An improved form of wick-plaiting machine is shown in Fig. 95; of a triple set, one apparatus only is seen. The contrivance is simple, but very ingenious. The strands of cotton yarn are carried on 3 revolving bobbins, whose gyrations are regulated by the beater beneath. The plaited wick is passed away in an endless rope over the wheels. In principle, this is identical with ordinary braiding machines, but it differs from them in always having but 3 bobbins. Given proper materials, the success of a wick depends upon the manner in which it is plaited, and especially upon the relative tightness of the plaiting. Stearin candles require a moderately tightly braided wick; for paraffin, the braiding must be extra tight; for sperm and wax, on the other hand, it needs to be unusually loose. Few candle-makers plait their own wick, at least in any quantity;

they prefer, in most instances, to entrust the work to cotton spinners who make it more or less a specialty.

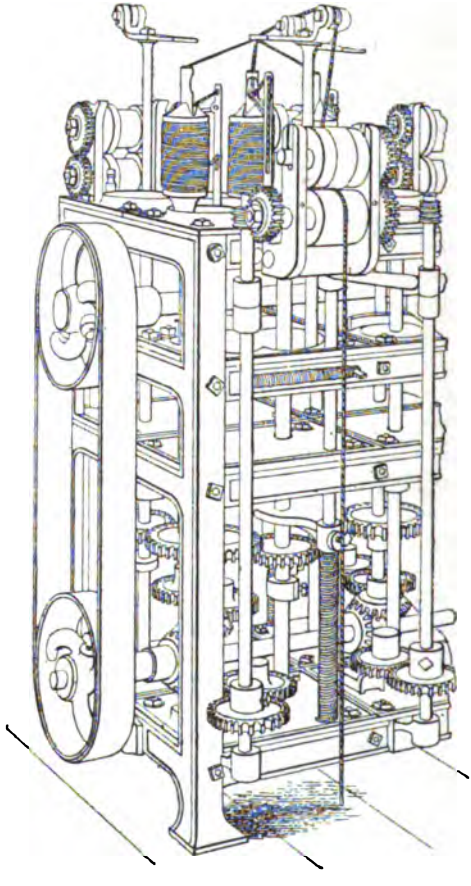


FIG. 95.—WICK PLAITING MACHINE.

After being twisted or plaited, the wicks are bleached in the ordinary way, and thoroughly dried. Before being used

by the candle-maker, they are dipped in a bath of pickling liquor, the effect of which is to retard combustion, and to help in causing the destruction of the ash. The pickle most commonly employed is a solution of about 1 lb. of boracic acid in 75 pints of water; in this, the wicks are soaked for about 3 hours. When taken out, they are either wrung, or put into a centrifugal machine, to remove the first excess of water, and are then completely dried in a tinned-iron box, provided with a steam jacket, or in a room heated with steam, with racks supporting shallow trays on each side. The bottom of the trays must be open, and may be a grid formed of $\frac{1}{4}$ in. or $\frac{3}{8}$ in. rods, placed at $1\frac{1}{2}$ in. distances apart, and the trays should slide on fillets fixed in the racks. Good ventilation will quicken the drying of the cotton, which should be laid on the grid crosswise. In a small factory the winding may be done by hand with a *swift* and reel, but in a large one it will be more economical to use power. The arrangement is simple. On the wall, small grooved pulleys are fitted to run in a frame, their axles projecting so as to form tapered spindles. The speed is got up by driving with a gut band off a larger grooved pulley, which in its turn is driven from a main shaft. The bobbin is fitted on the tapered projecting end of the spindle, tightly enough to serve for winding, but loose enough to slacken if the wick tangles. Two or three feet away from each spindle two small reels are erected on an upright also fixed to the wall. One of these is movable, so that any size of hank can be made to revolve over the pair. The operation of winding is very quickly done, and does not require description. Various other pickles are recommended; the principal are—(1) A solution of 5 to 8 *grm.* of boracic acid in 1 *litre* of water, to which 0·3 to 0·5 per cent. of sulphuric acid has been added; (2) a solution of ammonium phosphate (used in some Austrian works); (3) a solution of sal-ammoniac at 3°–$4\frac{1}{2}$° Tw. (2°–3° B.), proposed by Dr. Bolly; (4) a solution of 2 oz. borax, 1 oz. potassium chloride, 1 oz. potassium nitrate, and 1 oz. ammonium chloride in 3 qt. water; (5) the wicks of the

newly-introduced "snuffless dips" are plaited, and are then soaked in a solution of bismuth nitrate. (6) Another good solution, in extensive use, is the following:—sulphate ammonia, 1¼ lb.; nitrate of potash, ½ lb.; borax, ½ lb.; distilled water, 1 gal.; (7) J. L. Field treats wicks to prevent smouldering when extinguished, by steeping them in a solution of phosphoric acid, or ammonium phosphate, or ammonium phosphate and borax, or ammonium phosphate and boracic acid. (8) Duparquet manipulates wicks to prevent smoke and smell on extinction. Bleached wicks are soaked for ½ hour in a bath containing 16 *grm.* ammonium phosphate and 7 *grm.* sulphuric acid at 168° Tw. (66° B.) per *litre* of distilled water, the acid being added when the phosphate is well dissolved. Unbleached wicks require 40 minutes' soaking, and need a preliminary cleaning in a bath of 6½ oz. volatile ammonia, 1 oz. sulphuric acid at 168° Tw. (66° B.) in 246 oz. water, the mixture being boiled by steam, and the wicks kept in for 1½ hour, then boiled for ½ hour in pure water, rinsed in cold water, wrung, and dried, ready for the second bath.

To get the best results there must be a careful adjustment of the size of the wick to the diameter of the candle of which it is to form a part. The size nomenclature of candles is based on the number of candles required to make up a pound weight, modified, so as to indicate differences in length required to meet the popular demand. Thus there are 6's, short 6's, and short short 6's, and other sizes of the same weight, but of different diameter. The size of the wick also is designated by the number of threads in each fold of the plait. For example, 3–20 plait is a plait of 3 ply of 20 threads each; any departure from the usual size of the ultimate threads is indicated as special. In ordinary plait for common paraffin candles, sizes 1's and 2's will be suited with 3–18 plait, 4's and 6's with 3–14 plait, 7's to short 14's inclusive, with the exception of long 10's and long 12's, with 3–11 plait, and for these and for 16's to 20's and smaller sizes 3–8 plait will be found suitable. When a candle, protected from draughts, emits smoke in burning, the wick is too large for the quality

of the material used, and the adjustment demands attention. If a wick stands bolt upright during burning, it has either been imperfectly wrung, and excess of solution has gravitated during drying to particular portions of it, or by torsion it has been deprived of its elasticity. On the other hand, a wick which bends too low, making the edge of the cup to melt away on the one side, and thereby causing guttering, may be improved by strengthening the solution. Sputtering indicates wetness of the wick caused probably from an overflow of water in the candle-moulding machine. Dry wick feels crisp to the hand, and the hank when twisted and applied to the ear, will by an abundant crepitation give unmistakable evidence of dryness. Mere dampness of a wick does not cause spluttering, but the dampness will reduce its capillarity and reduce the rate of consumption, and consequently also its illuminating power.

When a candle, on being blown out, continues still to smoulder, burning down to a mere stump which is lit again with difficulty, a necessity is indicated for strengthening the solution. Borax or boric acid will give the necessary backbone, and ammonia salts, as the sulphates, chlorides and phosphates, also potassium salts will help to render the wick fireproof, except in the outer mantle envelope of the flame. This part of the flame is intensely hot, and here the carbon of the wick will be burnt up; its ashes will be fused into microscopic globules of glass by union with the salts, and carried away by the strong air currents produced by the heat. Weak acids are used to dissolve out earthy matters of the wick, in some cases, where an easier flow of the fuel is wanted. Each maker has his own ideas as to the best dressing required, and varies his pickle to suit the material. A wick that has been severely stretched seldom bends well. It remains in the axis of the flame, is unconsumed for lack of heat, the flame gets too large, and smoke is produced. Too sudden bending throws the flame out of its compact shape, it cools, and loses its intensity.

Having described the nature and preparation of the

materials which, in one form or another, constitute the two component parts of every candle, the next consideration will be the manner in which their combination is effected. Three plans are in vogue, each exceedingly simple; dipping, moulding, and pouring. The first is employed for common tallow candles, which are accordingly called "dips." The rods supporting the twisted wicks, as they come from the twisting and cutting machine, are transferred to a frame capable of being raised and lowered at will. This commonly takes the form of a beam, but a better arrangement is seen in Fig. 96. The frame, made of iron, and capable of revolving, is so suspended that a perfectly horizontal position is always maintained, even under undue pressure at either end; in this way are secured a uniform length of candle and a plumpness at the top, which is difficult of attainment, even by skilful workmen, by the ordinary beam. Under the frame are placed troughs containing melted tallow, into which the suspended wicks are repeatedly dipped. After each dipping, the adherent fat is allowed to cool sufficiently to retain a new coating on fresh immersion. The process is renewed until the candles have grown to the proper thickness; they are then left to cool and harden. Tallow dip candles are still largely manufactured, and are much employed in mines and small factories, and by domestic servants, as well as in cottages. They are also very suitable for artisans working in draughty places, as the wicks being large are not easily

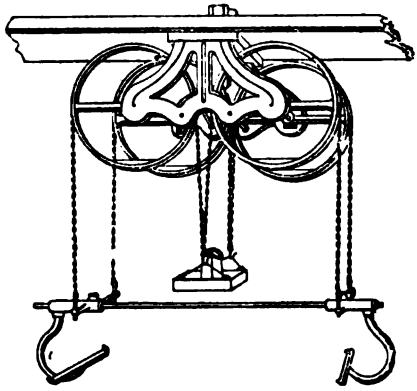


FIG. 96.—DIPPING MACHINE.

extinguished. They have been largely replaced for domestic use by the small moulded "cottage composites" made from distilled fatty acids, with a self-consuming wick. There is also a dip candle made from the same material, and in the same sizes as the old tallow dip, called the "snuffless dip," which, as a candle for carrying about, is superior to the paraffin candle. It is rather more expensive than the commoner paraffin, but, by the introduction of a core of softer material, the cost may be reduced. The first operation in the manufacture of these is to wind a continuous length of plaited and prepared wick around a frame of narrow, but stout, iron hoop, bent into the shape of an oblong rectangle. All the frames are of one length, but the widths vary to suit the length of the candle required. To the upper side of this frame is temporarily attached a lath or broach corresponding in length with the frame. The dipping is done at first three or four times, and when the wicks have acquired sufficient rigidity, a knife is passed along the lower side of the frame, cutting the wicks at the lower end. The iron frame is removed, and the coated wicks are left suspended on the broach. The broaches are about 3 ft. in length, and carry 40 candles each. The candles during the process of dipping are kept apart from each other by the width of the broach; and the ends of the four broaches are caught up, and kept separate, by holes made to fit them loosely in the blocks, which serve as handles wherewith to carry them, and which, in the operation of dipping, are laid on the supporting brackets of the dipping machine. Thus 160 candles are dipped at once, making 20 lb. of 8's, 16 lb. of 12's, and so on. The dipping is continued at intervals till the required weight is attained, and which is shown at once by the perfect balance. The material in the dipping trough is, by constant additions of warmer material, kept near the point of congealation. The plant required is inexpensive, consisting mainly of frames, some of them revolving on pivots, to carry the broaches with their candles while cooling, a wheel for winding wicks on frames, and a few melting tubs.

Pouring is used only with candles made of beeswax, which cannot be moulded, for the candles refuse to leave the moulds, or crack while doing so. The wicks are placed upon a horizontal hoop, and the operator, holding this with one hand, pours the previously melted wax over the wicks with the other. After three or four revolutions, that hoop is laid aside, and another is substituted. At a certain period the candles are reversed, as there is a natural tendency to thicken at the lower extremity. They are then rolled on a marble slab, under a weighted board, and are trimmed to the required length with a knife and gauge. A well-made wax candle should show rings like a tree, where the different layers have been superposed.

By far the greater number of candles now manufactured are moulded, by which they acquire a much more finished appearance. The most simple form of moulding machine is that known as the "hand-frame," which is in use among small manufacturers, and for particular kinds of candles, bought by those who, for reasons best known to themselves, prefer "hand-made" articles. The hand-frame contains from three to thirty-six mould-pipes (each making one candle), held together by woodwork, and opening into a trough at the top, their points being downwards. The wicks are cut to the proper length, and provided with a loop at one end, which is caught by a long crochet-hook, and thus one wick is drawn into each pipe, where it is secured by a peg at the tip, and a cross-wire at the "butt" end of the candle. The frames are then heated to a temperature about 10° F. (5½ C.) short of the solidifying-point of the candle-material, which is then poured into them in the case of fatty acids, as cold as possible, provided that there are no lumps of solid fat in the material. The trough is filled full, to allow for the contraction of the candle when cold, and the superfluous material is eventually removed with a straight-edged trowel. The hand-made candle is readily distinguished by the little groove which the wire wick-holder makes in its base.

The arrangement of the candle factory is of great im-

portance. The boiling-up department must be well ventilated, so that the steam rising from the vats does not pervade the rest of the building, and after condensing on the roofs, drop like rain on the goods. For economy of working, it should be as central as possible in relation to the moulding room. The vats should be set at such a height that the candle-material shall, by its own weight, fall or flow into the drying pans. They must be strongly built of wood, and hooped, and each heated by a perforated copper coil inside, around, and near its bottom. On the side of each vat, near the bottom, there should be a large brass cock for running off the wax, and, in the bottom, there should be a hole, fitted with a movable plug, for drawing off the water, and below, a separator, to conserve any wax which may escape with the water. This separator may extend under several vats.

No iron must come in contact with the wax after it has been mixed with the percentage of stearic acid which has been added in the vats. The drying pans, steam cased as they must be, for heating, may be of earthenware or enamelled iron, or, as they are in rare cases, silver plated. If these are placed in the moulding room, so as to do away with long hand carriage by the maker, the exhaust steam from them should be led away to a steam trap. The difficulty of connecting the drying pans to distant vats is the chemical one of getting a pipe or gutter of a metal or suitable material which shall not be affected by the fatty acid. This is sometimes overcome by keeping the stearic acid separate, and measuring it into the drying pans, but this method has disadvantages. Sometimes, for common candles, American scale is wrought along with semi-refined. It must be blended in the melting house, but the melting had better be done in the same way as recommended for scales for refining, at a point in the yard convenient to the stocks, where it can be done in an underground boiler, and blown into elevated settlers, whence it can be drawn as required. In blending paraffins of different melting points, they will be found to give a melting-point

the mean of the ingredients, but the stearic acid admixture will lower it, whatever be its melting point.

Coming to the moulding room, the steam and water systems of pipes should be so arranged that the supply to every part of the house can be controlled. The water supply should be ample, and pumped into a tank at a good elevation. It is not often that pumping can be avoided altogether, but an attempt should be made to secure such an outfall that a double pumping may be dispensed with. Cast-iron pipes for steam and water mains answer well, and service pipes with branches at 4 ft. distances, with flanges at each side, the steam service pipe above and the water below, feeding two rows of machines, set handle to handle, and with working distance between, is a good arrangement, as, in the case of cocks needing repair, all that needs to be done is to slacken the flange bolts, put in a diaphragm of sheet iron when the steam or water is off, screw up again, and go on as before, without interfering with the other machines.

From the flanges on each side of the steam and water service pipes depend, at the 4-ft. intervals, two malleable feed pipes. They are screwed into cast metal flanges, and bolted to the flanges, as just described. They are so bent at the top as to give sufficient working room between the handles of the pair of machines which are set one at each side of the service pipe. A steam and a water branch are connected to each machine by means of a bend and T-piece, and a strong brass stuffing cock on each side governs the steam and the water supply. Another and much neater arrangement is to have to an outward bend on the machine supply pipe a strong brass three-way cock, the dependent pipes, one on each side, with a bend, being coupled to each side of the cock, the machine being fed through the key, either by steam or water, according as the opening in key is turned to the steam or to the water side. Perhaps the former and usual arrangement is the more economical. Between each two rows of machines the passages should be roomy, and the waterways, of size proportioned to their duty,

should be covered. The candles have to be removed along these passages, and sufficient bench accommodation for newly made candles must also be allowed for.

The machine now usually employed for candle-moulding is an improvement by Cowles upon one introduced into this country from America about 1848–50.

It is shown in Fig. 97; A, standards and water-box, with 80–112 candle-moulds enclosed; B, movable clamps, for

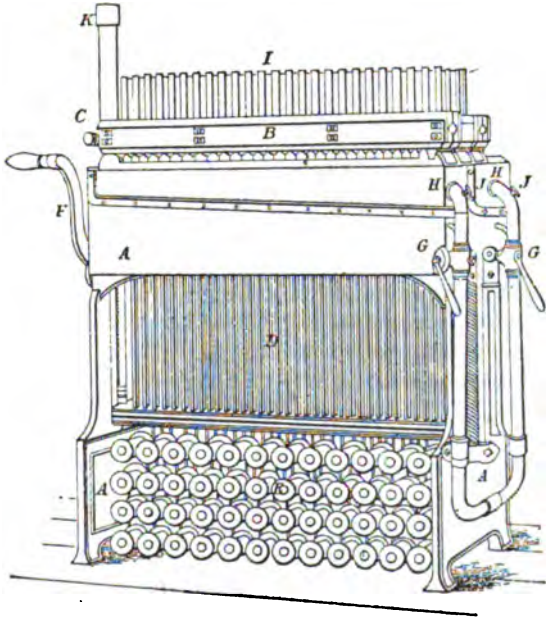


FIG. 97.—CANDLE-MOULDING MACHINE.

holding the ejected candles; C, handle of eccentric wedge, for opening and closing the clamps; D, pistons, having the tips soldered at the top ends, which are fitted to the lower ends of the candle moulds; E, cotton bobbins, revolving on strong iron pins; F, crank handle for raising the pistons, by

the action of which the newly-made candles are ejected into the clamps; G, handle of gun-metal gland cock, for emptying water-box; H, overflow pipe, which prevents the box from being overcharged with water; I, newly-made candles, held by clamps while the melted material is being poured in, so that the wick is centred in each mould; J, a clearing pin, to enable workmen to clear the bend of the overflow pipe if it should become choked; K, a pipe to admit hot or cold water to the water-box.

Essentially this is the candle machine of to-day, but by doing away with the side gutters, which in the illustration are shown riveted to the outside of the water-box, and providing for the carrying away of the hot water by an internal overflow into channels placed either along the sides of the water-box or between the filling troughs, or both, it has been made much more compact without detracting from its efficiency. The waste pipe has also been carried down the inside of the leg to the drain below, instead of outwards, as shown at H, still further economising space. By introducing the steam and water into the end of the water-box, near its bottom, a two-way gun-metal stuffing cock on each governing the supplies, the water mains and service pipes being kept below the floor level, a neatness and cleanliness is secured that cannot be got by the most methodical overhead system, to say nothing of the tangled forest appearance which some factories present, where every other interest has had to yield to that of present convenience.

The method of using the machine is as follows:—After having made the connection between the hot and cold water pipes and the machine at K, and having connected the outlet-pipe with a drain, the machine is ready for cottoning. The pistons are raised by turning the crank handle F until the tips are level with the butt ends of the tin candle-moulds, where they can be held by the pawl catching in the pinion. A fine wire, doubled, and of sufficient length to go through the tip-mould and piston, is then inserted, and extended below the piston sufficiently to enable the operator to pass

the candle-wick end through the loop. This permits the cotton to be drawn up through the mould; it must then be secured in any convenient manner during the first filling. The crank F is returned, the water in the machine is raised to about blood heat, by gently opening the valve on steam supply pipe near K, but not shown in the figure. The temperature is adjudged either by dipping the finger into an opening left over the water-box, or by applying the palm of the hand to its side. When the water is adjudged warm enough, the steam is shut off and the machine is charged. A liberal surplus of candle material is left in the trough to make up for the shrinkage in the moulds. The cold water is now turned on, and in 15 to 20 minutes, or more, the candles will be found solid to the core. After running a knife along the sides, and around ends of both troughs, the surplus is removed. This is usually done with a scoop fitting the troughs, but with parts of its edges removed, so as not to interfere with the candle ends. The ends of the candles are next finished with a sharp knife. Sometimes both operations are done with one sweep of a bevelled edge chisel. In both cases the cuttings have to be remelted, and judiciously blended with better material. The clamps B should be placed open over the machine; the crank-handle F is then turned, and the candles are ejected into the open clamps. These are then closed by the handle C, so that each candle is held in its proper position. The crank-handle F is then returned to allow all the pistons to recede into their places: the wicks are thus held in a central position by the candles I and the cotton-bobbins E. The cotton should be slightly strained under the piston plate. After again warming up the machine more melted material is poured into the moulds to form a second batch; when these are nearly set, the wicks are severed under the clamps by scissors, or by one stroke of a very sharp knife along each row of wicks, and the first batch is removed in the clamps. From the clamps the candles are emptied into boxes about 4 ft. long, 1 ft. wide, and 14 in. deep, without top or front. Each of these boxes

if intended to be removed to the candle-house, should be tared and its weight marked on it, so that nett weight can be ascertained and credited to the maker when the goods are passed over the weighing machine, as they are sent on to the packing rooms. With plain end machines, averaging all sizes, one man in 9 hours with 6 machines will turn out 120 doz. lb. of paraffin candles; and with the aid of a boy with nine, will turn out 168 doz. lb. Each machine in the double shift should on an average turn out from 28 to 30 doz. lb. In these cases the material is made ready and to hand. With ordinary self-fitting machines, not half these quantities will be got.

The candle-packing room is provided with benches at which the folders work, arranged either in parallel rows or otherwise, but with sufficient space reserved for candles awaiting attention, for working room, and for passage. On the bench, immediately before the folder, is the folding block, and before him, a little to the right, is a protected gas jet for melting the stick of sealing wax, or a heated vessel containing the wax in a melted state. Behind him are the candles, loose, in covered boxes, piled up, or in the trays before described, supported on trestles. The moulding block is capable of adjustment to any size of packet required, and holds the candles in the hexagonal position they are to take in the packet. The folder takes up the outer wrapping sheet, lays on it a sheet of lining paper, both cut to size, and lays them in his block. With the almost unerring accuracy which practice gives, he lifts the exact number of candles required for the packet, lays them on the paper in order, folds the paper around them, and seals it, lifts the packet out of the block, folds the paper over the candle butts, and then over the tip end, seals both ends and puts the parcel on the pile in less time than it takes to describe it. Such facility does practice give that some men dispense with the block entirely, and, amid a great variety of sizes, can pick up the exact number to form a 3 or 6-lb. packet without a mistake. A good folder will tie up in packets the contents of 40 six-doz.

cases per day, or 240 doz. lb. The candles are built up on the bench in the case of 3-lb. packets, in piles of 24 packets each, labelled and passed on to the packer, then to the nailer-up, and finally to the warehouse.

Self-fitting candles are usually packed up by the makers immediately they are removed from the clamps, and passed on to the packing room for nailing up; and export candles are put up in cartoons of 6 candles each, which are generally packed in boxes holding 25 packets of every conceivable weight. These a man can do at the rate of about 100 cartoons per hour.

The temperature of the water in the machine is easily regulated, by shutting off or admitting hot or cold water, as required, at the T connection at K. The internal immersion pipes, situated inside the water-box and between the rows of moulds, are perforated. These machines occupy only about 3 ft. × 2 ft. of floor space, and are made to mould candles from 1 lb. each to 56 to the lb. These extreme sizes are used for church purposes and for Christmas tree decoration.

A candle machine may be made with moulds of different sizes on each side. This may be useful in very small works, if the sizes are in equal demand, but generally it is not a good plan, as the demands for certain sizes fluctuate. Different lengths may be got from the same machine by "blocking up." This can only be done to a limited extent; that exceeded, there will be leakage past the pistons, and a risk incurred of clogging the wicks with the candle material, which will prevent the wick running through the holes in the pistons, which are purposely made small to give due tension to the wick. There are also reasons connected with the wear of the machine, which render it inadvisable to exceed the demands of necessity in this direction. In order to secure rapid cooling, the pipes are made very thin, and even when made of the best material, can easily be rendered useless by forcing. It will be found better to pay a little more as wages for the candlemaking, and to encourage the candlemakers to take care of the machines, than to have them spoiled by rash and careless working.

A great improvement in the candle was made in 1861, when J. Lyon Field patented the conical "butt" by which a candle could be adapted to any candlestick, without paper or scraping. The moulds for the butts used to be cast in a separate frame, which was removed when the candles were finished, by a chain and pulley, and the candles were then pushed out in the usual way. Several different patents exist for accomplishing the same object more neatly. A newer patent of E. Cowles, Novelty Works, Hounslow,

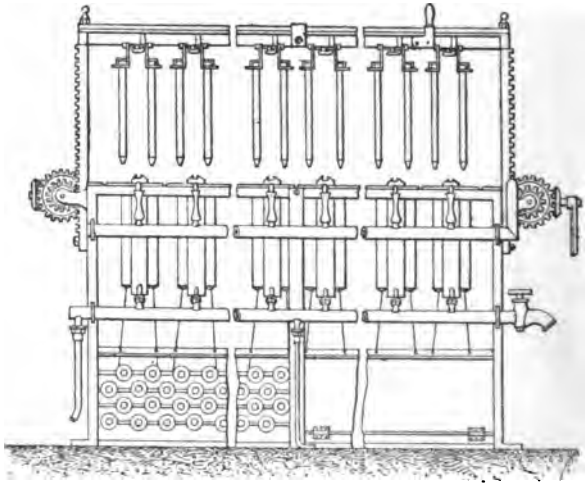


FIG. 98.—CANDLE-MOULDING MACHINE.

raises the bed containing the conical butts, and divides it while rising, thus obviating great loss of time and wick; in fact, enabling conical ends to be made as easily as plain ends. Some novel features in candle-moulding machinery have also been introduced by J. and W. Barlow, Rochdale.

Another form of moulding machine is shown in Fig. 98. It is manufactured by Galabrun Frères, of Paris, and is in general favour on the Continent; it is said to be capable of

turning out 200 candles per half-hour; but it is only suitable for stearic acid, or similarly hard material.

When moulded and cold, the candles are taken to little tables, fitted with circular knives revolving at high speed.

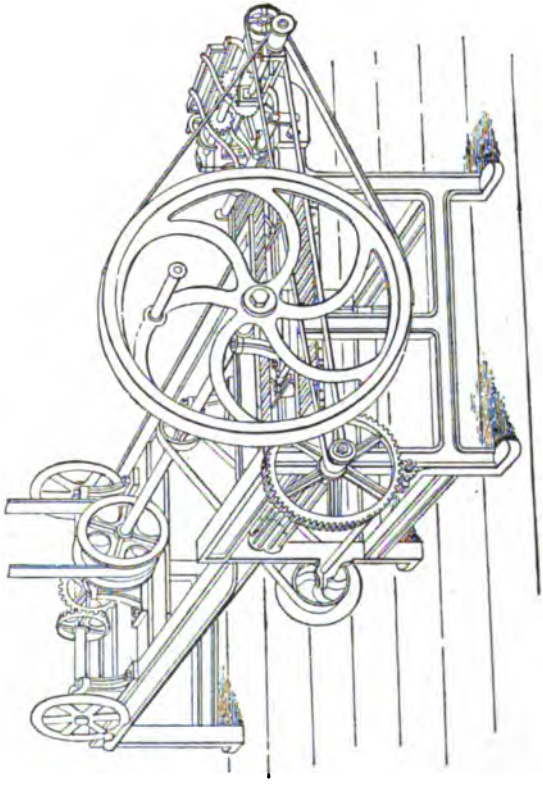


FIG. 99.—CANDLE TRIMMING AND POLISHING MACHINE.

Here the butt ends are trimmed, and the length of the candles is adjusted to the weight. Some of the superior kinds of candle undergo a special polishing operation, performed by subjecting them to the friction of felt and other substances.

In Fig. 99 is seen a trimming, washing, and polishing machine combined, as made by Galabrun Frères.

The various fancy patterns of candles, rope-like, &c., all require special hand-moulds. Spiral candles are sometimes made plain and afterwards turned in a lathe. Mr. Hugh Findlay, in a machine similar to that shown in Fig. 100, by means of very accurately made moulds, makes spiral candles direct. The section is an end one, and is a good illustration of one variety of self-fitting candle machine. The distribution of the water is well shown, and the candle butts are kept well down for rapid cooling. The caps being solid in this case necessitates catching up the wicks before filling. In some machines this is avoided by making the caps to open with a hinge.

In addition to the candle-machinery makers already mentioned, Messrs. Biertumpfel & Sons, 138 Albany Street, Regent's Park, have long been special makers known to the home and colonial trade.

The devices to prevent guttering are endless; one which answers well for the upper half at any rate, of the candle, is to mould it partially hollow, by inserting 2 or 3 solid rods in the mould, and withdrawing them when the candle is solid. J. K. Field makes lateral apertures near the base of the candles to admit air for passage through longitudinal apertures, so that the air issuing near the flame may perfect the combustion. A. Field makes them with spiral passages to receive surplus melted material and perfect combustion. De Lastelle arranges a number of wicks in a circle, and leaves a hole through the centre of the candle to admit air. Joly makes the wicks with independent core pieces, to perfect combustion and obviate snuffing. Greaves prevents guttering by making the candle fluted externally. Lyttle attaches an incombustible base. Farrow places an incombustible obstruction in the wick, which causes extinction; the remaining end is then inverted and lit by the wick projecting from the base. Griffith places a metallic cup at the base, so that the final portion of the candle burns as a night-light.

"Disinfectant" candles are those which contain sub-

stances capable of emitting antiseptic or deodorising vapours during combustion, such as carbolic acid, sulphur, organic sulphides, camphor, eucalyptus oil, cajeput oil, &c.

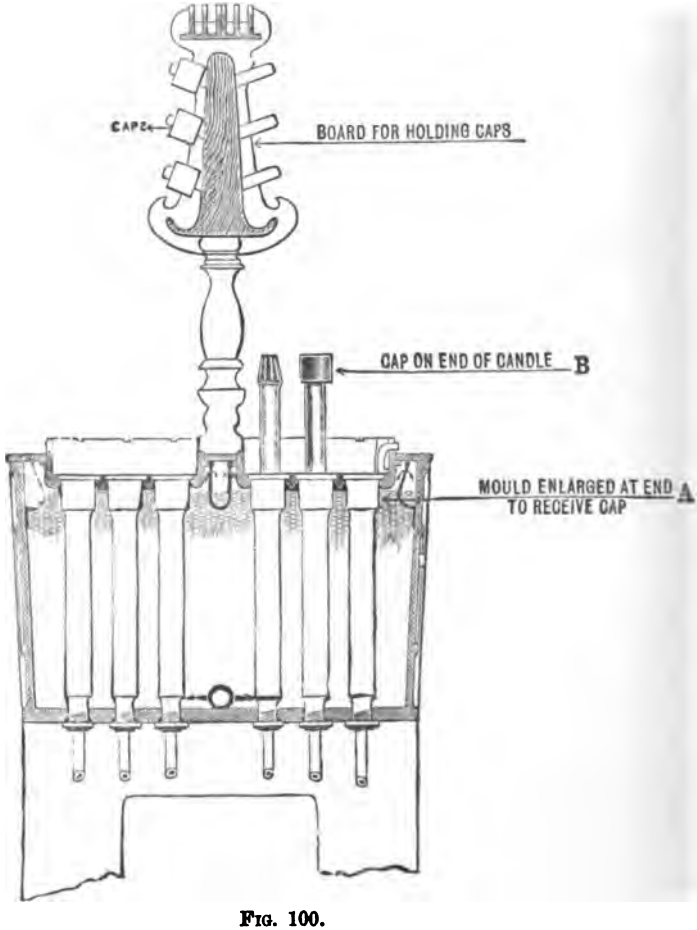


FIG. 100.

For colouring candles, vegetable or anilin dyes are almost always used; mineral dyes are to be avoided, as they seriously

interfere with the combustion of the candle. In order to tint paraffin candles with many dyes which are insoluble therein, the dye must first be dissolved in stearin, and then that compound will be readily taken up by the paraffin.

As an illustration of the extraordinary variety of candles which is required to meet the demands of daily life, and the caprices of fashion, it may be mentioned that Price's Patent Candle Co. have in use, moulds for the direct production of no less than 400 different sizes of candles, that is taking no account of requirements of customers for other sizes which can be met by cutting down. But as the Company makes candles of 60 different qualities, dependent on variations in actual material or in colour or hardness, it is evident that they are in a position to produce an almost unlimited variety of size and quality of candles. As a matter of fact, they are called upon in the ordinary course of business to supply nearly 2000 kinds, and find it necessary to keep about 400 different kinds regularly in stock.

In moulding candles of any kind, great attention must be paid to the temperature of the melted material, to the warmth of the mould, and to the rate at which the mould is cooled, in order to produce the best possible appearance. If the mould, or the candle-material, or both, are too cold, the surface of the candle will have holes, owing to the air bubbles not having had time to escape, and wavy marks on its surface will indicate plainly that the material has congealed immediately it has come in contact with the metal. Moulds with high internal polish, also kept clean on their outside by frequent cleansing of the water-box, if dirty water is used, material at 170° F. to 180° F. (78° to 82° C.) poured into a warm machine and cooled quickly will secure a high polish on a paraffin candle. In the case of a stearin candle, the material must be on the point of congelation, and the water in the machine must be kept warm, and merely tempered down by the addition of a little cold, as required. Making the machine too hot, on the other hand, moulds clogged on the outside by the dirty sediment from the water, which reduces the cooling power of the water, will ensure a

greasy, scaled and dull candle. Mottled candles are produced by the use of a wax containing free oil; the addition of 4½ to 5 per cent. stearic acid will remove this objectionable appearance.

Night-lights.—The making of night-lights is an important branch of candle manufacture. Clark's patent "pyramid" has an enormous sale. There are two distinct forms of night-lights made, namely, those which are either poured into a paper envelope, or moulded first and then fitted into it, which are intended to be placed when burning in a saucer of water, and those which are moulded for the purpose of being burned in a small glass tumbler for home use, and the bucket light for outside ornamental illumination. Paper and cardboard have now entirely supplanted wooden shavings for the manufacture of boxes and casings, and at present, in that department at Belmont Works there are 150 girls employed.

The wicks for night-lights are, to give them firmness, sometimes plaited around a core of straight thread of the same cotton. They are then chemically treated as in the case of candle-wick, and after thorough drying are drawn through melted beeswax, and through a small gauged hole in a warm iron plate. The other tools required are two tin drums, which are alternately carried to the opposite ends of the room, and made to revolve on spindles fixed on the wall. The wick is wound off the one and on to the other till sufficient wax coating is got, and then it is wound on a reel about 3 ft. 6 in. long; a knife cut at each end of the reel severs the hank, the ends are reversed so as to put the structure of the wick in one direction, and it is cut into the required lengths on a board fitted with a gauge to guide the knife. Before being cut the bundle of wicks is tied at short distances, so that each piece after being cut is a tied bundle, and the end intended to be cemented to the bottom of pill-box, with sustainer above, is dyed with a little anilin-tinted stearin.

Night-lights of paraffin wax are moulded in a machine specially made for them, but very much similar to that figured on p. 383. Instead of a crank for lifting the candles a simple lever is used; and instead of the machine being fitted with

bobbins, it has a special plate supporting wires which occupy the position, during moulding, that the wicks are to do afterwards. The envelopes may be purchased from any pill-box maker, ready, for about 4½*d.* per gross, the ornamental bands being supplied to him. These will cost about 8½*d.* per 1000. The sustainers will also cost about 6*d.* per 1000. A boy takes up a wick, dips the dyed end in a small vessel containing a cement made of rosin and Carnauba wax, after having threaded the sustainer with it, and fixes the end in the centre of the bottom of the pill-box. Another takes up the short candle, clears the hole with a steam-heated piercer, adjusts the wick in the hole, and pushes the candle over it into its place in the box. The melting-point of the material, which must be very pure, must be adjusted, else the light being small, it may fail to melt the material from the sides of the box and be extinguished for want of fuel. The wick must be firmly fixed so that it shall not fall over when burning and drown, or scorch or set fire to the casing.

The "New Patent" night-lights differ from the foregoing, not only in being made from much better materials, but also in the method of manufacture and mode of burning. Cases are dispensed with, and the fatty material, usually derived from coconut, is moulded to the required shape by being run into a frame, which consists of a number of moulds or cups securely fitted to a bed of iron or wood. Into these the melted material is poured and left to cool. When cold, the excess of fat is scraped off with a blunt tool, and the night-lights, ready punctured for the insertion of the wick, are lifted out by a screw. On each wick is threaded a tiny square of tin-foil, which is to serve as a support for it during the later stage of the combustion of the light; the wick is then thrust through the little disc of opaque white fatty matter, and is secured by a cleat effected by a sharp blow on a miniature vertical anvil. The rapidity and precision with which the lads perform this operation is something to admire. The lights are now ready for burning, for which purpose they are placed without water in little glass cups. Night-lights are made of various sizes, calculated to burn

for 6, 8, or 10 hours. Each box of 6, 8, or 10-hour lights, contains 12, 10, or 8 respectively, the rate of burning being for paraffins about 43 grains per hour.

Payne and Freestone make night-lights with a paper bottom above a plaster filling, fixing the wick out of the centre of the paper, to ensure its perfect combustion by inclining to one side; they make the wicks of inkle, with a wire running through to regulate the position, and gimp the inkle and wire together. Clarke makes them with a plaster casing having a concave bottom, to ensure complete combustion. Berta makes the cases in one piece, and fixes the wick upright till all the material is consumed.

ILLUMINATING VALUE.

It is somewhat remarkable that purchasers, in judging of the value of a candle, are entirely guided by its mere appearance, and more particularly by its colour. The primary object of a candle is to give light. In estimating the value of any light-giving material, 3 factors have to be taken into account, viz.:—(1) the cost of the material; (2) the rate of its consumption; (3) the luminous intensity produced. In practice, in this country, a sperm candle burning at the rate of 120 gr. an hour, is taken as the standard light with which all others are compared. This is the explanation of the phrases "16-candle gas," electric light of so many thousand "candle-power," and so on. Such a standard, however, is unsatisfactory at best, since very slight inequalities in the wick, or changes of its curve in the candle flame, materially affect the luminous intensity, without appreciably altering the amount of sperm consumed. Various other standards are in use in other countries, and the values of each are convertible into terms of the others. 100 sperm candle power, as before defined, is equal to 1023 of the German-paraffin candle standard, or to 1248 of the Hefner lamp, or to 104 of the Paris Carcel lamp. As presently the illuminating value of the Carcel lamp will be referred to, it may be noted that

this lamp is an Argand, burning refined rape oil, at the rate of 42 grm. per hour, any variation between 40 and 44 grm. having to be calculated for as in the case of the standard sperm; any further departure from the normal rate renders the results untrustworthy. The weight of the wick, the height to which it is trimmed, as well as the position of the constriction of the chimney, have each an influence on the light value, and are regulated by statute. It is found that with all the standards absolute uniformity is unattainable, and the results are, even with the greatest care taken, only more or less approximate.

There are three variable elements, each of which must be duly considered. If, for example, paraffin and stearin (or composite) candles give equal light photometrically, and 1 lb. of stearin candles lasts 48 hours, while 1 lb. of paraffin candles lasts 54 hours (146 and 129 gr. per hour respectively), it is obvious that if the stearin candles cost 8*d.* per lb. and the paraffin candles cost 8¾*d.* per lb., the paraffin is really the cheaper of the two, and at 9*d.* per lb. would cost the same.

Some writers throw this calculation into the form of "cost per 100 of light," where the 100, or standard, is taken from a standard "hot-oil" lamp, burning every hour 815 gr. of oil, or 0·1164 lb. at 11*d.* a lb. Hence, in this case, the cost per 100 of light is $0 \cdot 1164 \times 11 = 1 \cdot 2804d.$ Comparing this with a wax candle, burning 125 gr. an hour, giving only $\frac{1}{11}$ the light of the lamp, and costing 2*s.* 6*d.* or 30*d.* a lb., we have as the cost per 100 of light:—

$$125 \times 11 = 1375 \text{ gr., or } 0 \cdot 1964 \text{ lb.} \times 30 = 5 \cdot 892d.$$

Proceeding in this manner, Dr. Frankland has drawn up the following table of illuminating equivalents, or the quantities of different illuminating materials necessary to produce the same amount of light:—

Young's paraffin-oil ..	1·00 gal.	Sperm candles	22·90 lb.
American petroleum, No 1	1·26 „	Wax „	26·40 „
„ „ No. 2	1·30 „	Composite „	29·50 „
Paraffin candles	18·60 lb.	Tallow „	36·00 „

	Price per lb.	Intensity of light in Carcels.	Intensity of light in standard sperm.	Grms. material consumed per hour.	Illuminating power = 100 Carcels.	Price of 100 grms. illuminating material.	Cost of the light per hour.	Cost of light of the same intensity = 100 Carcels in pence.	Do. in shillings and pence.
CANDLES.	*d.*							*d.*	*s.* *d.*
Fine paraffin	8	0·14	1	8·	73·5	1·763	0·141	100·71	8 4·7
Stearin	6	0·14	1	11·29	52·0	1·322	0·1492	106·57	8 10·6
Tallow	5	0·14	1	15·48	37·9	1·102	0·1706	121·85	10 1·8
Beeswax	36	0·14	1	11·35	51·8	7·936	0·9007	643·35	53 7·3
Spermaceti	26	0·14	1	9·85	59·7	5·713	0·5627	401·93	33 5·9
sp. gr. OILS. per gal.									
·881 Sperm .. .. 5*s.*	6·81	1·04	7·43	31·24	139·8	1·501	0·4689	45·08	3 9·1
·921 Colza .. .. 2*s.* 6*d.*	3·25	1·04	7·43	36	121·3	0·716	0·2577	24·87	2 0·9
·921 „ in ordinary moderator „	3·25	1·06	7·57	42	106·0	0·716	0·3007	28·39	2 4·4
·921 „ „ „ „	3·25	0·94	6·71	46	85·8	0·716	0·3293	35·05	2 11·3
·921 „ in *Carcel standard* „	3·25	1·00	7·143	42	100·0	0·716	0·3007	30·07	2 6·1
·802 *Mineral*, Scotch No. I. .. 6¾*d.*	·841	2·68	19·14	62	181·5	0·185	0·1147	4·28	4·3
·802 „ „ crystal .. 7*d.*	·872	2·41	17·21	62	163·2	0·192	0·1190	4·93	4·9
·817 „ „ No. II. .. 6*d.*	·734	2·35	16·79	62	159·1	0·162	0·1004	4·27	4·3
wick									
·800 (13 mm.) Amer. petrol. .. 7½*d.*	·937	0·81	5·78	20	170·1	0·2065	0·0413	5·09	5·1
·800 (50 mm.) „ .. 7½*d.*	·937	2·13	15·24	62	144·3	0·2065	0·128	6·01	6
·800 (2, each 25 mm.), Amer. petrol. .. .. 7½*d.*	·937	2·07	14·21	63	138·0	0·2065	0·1301	6·23	6·2
·800 (Amer. lamp, no chimney), Amer. petrol. .. .. .. 7½*d.*	·937	1·82	13·00	52	147·0	0·2065	0·1074	5·90	5·9
·800 (Round burner, 23 mm. dia.), Amer. petrol. .. .. .. 7½*d.*	·937	1·06	7·57	28	159·0	0·2065	0·0578	5·44	5·4

Taking into account the present market value of these various materials, the comparative cost of the light equal to that of 20 sperm candles each 1200 grains in weight (in all 24,000 grains) would be:—

	s.	d.		s.	d.
Young's paraffin No. 1 oil	0	1	Wax candles	11	10½
American petroleum, No. 1	0	1½	Composite or stearin do. ..	2	2½
Paraffin candles	1	10	Tallow candles	2	3
Sperm "	8	1½	Coal-gas	0	1½

The table on p. 397 is an attempt to put in concise form the approximate value of various illuminants, the intensity and rate of burning being chiefly taken from Baillé and Fery,* and also from other published sources. In comparing data, differences of intensity equal to fully 10 per cent. have been found for material nominally the same. The prices are approximate to those of to-day, but in the case of standard sperm candles, where the retail price is 4*s.* per lb., 2*s.* 2*d.* has been substituted, as there is a good margin for cost of manufacture at that price, spermaceti costing less than 1*s.* 6*d.* per lb.

As showing further, the influence of lamp construction on the light-giving efficiency of petroleum, and at the same the relative value of common coal-gas to it, the table on p. 399, taken from Leopold Field's Cantor Lecture, is appended.

Lewis Thompson gives the equivalent of 1000 cub. ft. of 13-candle gas as 44·$\frac{4}{7}$ lb. of sperm candles, so that 120 grains of sperm is equivalent to 0·3846 cub. ft. of 13-candle gas.

Although it is impossible to avoid the inference from the figures given, that candles in any form are very expensive illuminating agents, compared either with coal-gas or paraffin oil (or any of its numerous modifications), nevertheless the numerous advantages which they possess render it exceedingly unlikely that they will, to a greater extent than at present, be superseded by either of the two cheaper methods of illumination. Neither coal-gas nor paraffin oil can be employed except at what are, for all practical purposes, stationary points; they cannot be carried about, while

* 'Électricien,' 1889.

Description of Lamp Argand Burners.	Illuminating power expressed in sperm candles.	Illuminating power as expressed in cubic feet of common coal gas = 15 candles.	Hourly consumption of oil in grains.	Cost of oil for one week's consumption of 42 hours.	Cost of gas at 3s. 9d. per 1000 cubic feet for one week's consumption of 42 hours.	Proportional cost of gas and oil.
				d.	s. d.	
No. 1A burner wick	14	4·66	602	8·18	8·8	1 : 0·93
No. 1 burner (original), wick $\frac{7}{8}''$ to $\frac{15}{16}''$	15·67	5·22	864	11·8	9·86	1 : 1·21
Ditto, 2nd experiment	15·47	5·15	787	10·7	9·73	1 : 1·11
Ditto, altered	23·24	7·74	795	10·8	1 2·63	1 : 0·738
No. 2 burner (original), same size as No. 1 (of French construction)	14·93	4·98	818	11·1	9·4	1 : 1·18
Ditto, altered	21·52	7·17	788	10·7	1 1·55	1 : 0·79

evolving light, from one place to another, and are altogether destitute of that element of portability which renders the candle so valuable. Further, except in so far as a fire may result from actual contact between a candle flame and any inflammable substance, candles are absolutely safe illuminating agents, and persons burning them are not liable to the alarming explosions and fires which result from the careless use of gas or of paraffin lamps.*

It will be observed in the case of the oils that the illuminating value depends much on the quality of the lamp used, an inferior lamp showing a much lower value. The following statement concerning improvements made on lighthouse burners shows the importance of correct construction. Previous to the introduction of the Doty burner, and the use of mineral oil in lighthouses, the flames got from colza oil

* Valuable suggestions on this subject will be found in a lecture on "Accidental explosions produced by non-explosive liquids," by Sir F. Abel, C.B., F.R.S., delivered at the Royal Institution on March 13, 1885. and reported in 'Nature,' Nos. 803, 804, 805.

were of a power of, in standard sperms, for the 4-wick concentric burner, 270, for the 2-wick burner 58, and for the Argand 14 candles. The consumption of oil was such that for the 4-wick lamp one gallon equalled a consumption of 21½ lb. of sperm; in the 2-wick lamp it equalled 17½ lb., and in the Argand 14 lb. Captain Doty's improvements, together with the use of paraffin oil, raised the light to 288, 80, and 24 candles severally, and the oil consumption equivalents were 27½ lb., 27 lb., and 27 lb. Another improvement introduced by Farquhar gave:—

	6 wicks.	4 wicks.	2 wicks.
Burning colza oil	750	334	90 candle-power.
Burning paraffin oil ..	700	306	85 "

The following more recent results from the Trinity House burners, as improved by J. N. Douglas, are recorded:—

Illuminating Power.

	6 wicks.	4 wicks.	2 wicks.	Argands.
Burning colza oil ..	722 candles	328 candles	82 candles	23 candles
Burning paraffin oil ..	730 "	330 "	80 "	25 "

Equivalents of Sperm to 1 *Gallon of Oil.*

	6 wicks.	4 wicks.	2 wicks.	Argands.
Burning colza oil	26·76	28·10	25·77	28 00
Burning paraffin oil	24·98	26·94	27·43	28·57

The candle in performance does not appear to be susceptible of like improvement by altering its form. Not only does the light given by it fall very much below its value as indicated by the carbon it contains, and of what its constituents would give if burned in a well constructed lamp, but the material itself is much more costly per lb. than oils either fatty or mineral.

It may be remarked, in passing, that the difference in favour of gas, as against candles, is very much reduced in practice, since users of gas always habituate themselves to a much more intense light than when they employ candles. Probably the same thing will obtain, *mutatis mutandis*, when domestic electric lamps are substituted for gas.

Before proceeding to describe the various kinds of photometers, it may be useful to consider briefly two remarkable results of careful photometric observation which have an important practical bearing upon the most economical arrangement of a number of separate lights, whether they be candles, lamps, or gas-flames, i. e. the arrangement which will give the greatest total amount of light from the various illuminating sources.

The first is that when the flames of two lamps or candles touch each other, the luminous intensity of the combined flame is greater than the sum of the intensities of the separate flames. This effect was first observed by Dr. Benj. Franklin, and appears to be due to the increased temperature at the part where the flames overlap.

The second result may be thus expressed:—A comparison of the amounts of light afforded by the same number of flames in different relative positions proves that flame is perfectly transparent, and thus that the luminous effect of a row of lights is the same whether their arrangement is parallel with or perpendicular to the direction of the rays; similarly, a flat gas-flame gives the same degree of light in all directions.

The chief forms of photometer will now be described. The simplest, most readily constructed, and most easily used, is that known as Rumford's. It consists merely of a black cylindrical rod mounted vertically upon a stand or foot, and of a white screen upon which to receive the shadows of the rod. The lights to be compared (all others should be put out) are placed about until the respective shadows cast by the rod are of equal depth. The distances of the lights from the screen are then carefully measured, and each number thus obtained is multiplied by itself. The proportion between these products represents the relative intensities of the lights under examination. For example, suppose lamp A at 21 in. and lamp B at 30 in. from the screen gave equally deep shadows, then, since $21 \times 21 = 441$, and $30 \times 30 = 900$ lamp A is to lamp B as 441 to 900, or nearly as 1 to 2, or, in

other words, lamp B gives twice as much light as lamp A. As a similar calculation has to be made in all photometric tests (though it is frequently assisted by previously constructed tables suited to each instrument), it will not be repeated.

Bunsen's photometer depends on the equal illumination of two surfaces, and is much more exact than the preceding. The principle of it, with very slight modifications, is adopted in the delicate photometers used in gas-testing. The essential part of it is a piece of thin paper stretched in a frame, and the paper is rendered semi-transparent by being saturated with a solution of spermaceti in turpentine oil, with the exception of a central spot about 0·75 in. in diameter, which is allowed to remain opaque. In using it (in a dark room), the standard light is placed behind the spot, and the variable one in front. When the two surfaces are equally illuminated, the opaque spot disappears, and the whole surface of the disc is perfectly homogeneous in appearance.

Wheatstone's photometer consists of a small silvered polished bead, mounted upon a stem to which a looped motion is given by appropriate clockwork. When it is placed between two sources of light, and the clockwork is set in motion, two looped curves of different brightness are seen, so very close together that their intensities can readily be compared; the lights are then adjusted to give curves of equal brightness, and their respective distances are read off. The formation of a luminous curve by a moving bright bead depends, of course, upon the well-known principle of "persistence of vision," the simplest illustration of which is the circle of fire traced by a lighted stick whirled round by hand.

Letheby's and Evans's photometers are similar in construction, and both depend upon the principle of Bunsen's. Letheby's consists essentially of a long bar, at each end of which are supports for a light, one being the standard candle upon a Keate's candle-balance. Upon the rod slides a box, with holes on each side and in front; it contains the semi-

transparent paper with the opaque spot.* The box is moved until the spot disappears, when a pointer attached to the box indicates on a scale the intensity of the unknown light in terms of the standard. Evans's is a similar instrument, but the box is fixed, and the lights move along the bar.

In the concluding words of Leopold Field's Cantor Lectures:—"Gas and electric lighting have taken high rank as sciences, and justly so, involving as they do such profound thought and supreme skill. But the candle and the lamp have also called forth the highest powers of great scientists to perfect them—Chevreul and Young laboured for little else—and they are now nearly perfect of their kind. So, while honouring to the full the authors of the greater lights, we must not forget the many workers whose labours have brought the candle from the crude, smoky wax and tallow, to the beautiful form and light it now owns—workers of whom more than six generations sleep in the shadow of old Lambeth Church [London] with no other monument than that of a fair life and faithful toil!"

* Such discs may be procured in quantity from Sugg & Co., Charing Cross, London.

CHAPTER XVI.

GLYCERIN.

FEW things in the history of chemical industry are more wonderful than the enormous development in the use of this substance, which, a few years ago, was thrown away as a waste product, but which now finds so many useful applications in the arts and sciences. The researches of Chevreul, which demonstrated the constitution of fats, showed that glycerin exists in nearly all neutral fats (see pp. 4–11) in a combined state, and small traces of it have lately been discovered uncombined in palm oil. It is formed, as Pasteur has shown, in the process of fermentation, 100 parts cane sugar forming 3·5 parts of glycerin. Recent researches have also made it clear that its compound with phosphoric acid is the starting-point of a number of complex constituents of the brain. For practical purposes, however, glycerin is always obtained from the bye-products of candle-, and quite lately, of soap-factories. Cap worked out the first process for preparing it on a commercial scale from the waste liquor of the saponification of tallow by lime, in the first stage of stearic acid making (see p. 350). Early in 1854, Tilghman produced it by pumping an emulsion of 2 parts tallow and 1 part water through a coil of pipe heated to 612° F. (322° C.), after which the emulsion separated into two layers, the upper one of fatty acids, and the lower one of glycerin and water.

Several modifications of this were afterwards patented, but the only one worked on a large scale was that of G. F. Wilson and G. Payne, dated July 24, 1854, under which considerable quantities of glycerin have been made by Price's Patent Candle Co. In this process, neutral fats are put into a still

provided with a fine steam-worm, and with a fractional condensing apparatus of large surface, similar to that described on p. 356; they are then heated to 550°–600° F. (288°–315° C.), and plenty of superheated steam is injected—when mixed vapours of fatty acids, glycerin, and water are carried over to the condenser, where the divisions nearest the still collect only fatty acids, while those farthest from it yield mixtures of fatty acids with glycerin and water in various stages of concentration. Glycerin so made can be concentrated in a vacuum-pan. Care must be taken that the temperature does not exceed 600° F. (315° C.), and that plenty of steam is present, else some of the glycerin is decomposed, and acrolein, a compound most irritating to the eyes, is formed—

$$\text{Glycerin} = \text{Water} + \text{Acrolein}$$
$$C_3H_8O_3 = 2H_2O + C_3H_4O$$

Raw glycerin was also prepared from the water employed to wash the fatty acids after acidification (p. 356) of the neutral fats. The acid liquid was neutralised by barium or calcium carbonate, either of which was added until effervescence ceased, or by milk of lime: it was then concentrated to 48° Tw. (28° B., or 1·240 sp. gr.), in an open, shallow, cast-iron pan. Of late, however, glycerin has become sufficiently valuable to cause candle manufacturers to adopt that method of preparing fatty acids which gives them the greatest yield of glycerin from neutral fats. In this process, as patented by De Milly on Nov. 19, 1856, a copper vessel called an autoclave is employed; it is now very extensively used for glycerin making, both on the Continent of Europe and in England, and is thus conducted. About 1 ton of fat, usually mixed tallow and palm oil, is heated with 2 per cent. lime and ⅓ the fat-volume of water in an upright Papin's digester to 8 atmos. pressure for 4 hours, a little steam being allowed to escape at the top, so as to promote the agitation of the mass. The whole is then blown out into a tank, and the "sweet-water" is run off. The lime soap is decomposed in the usual way with sulphuric acid, and the resulting fatty

acids are either pressed for saponified stearin, or acidified and distilled for "distilled stearin." The "sweet-water" is

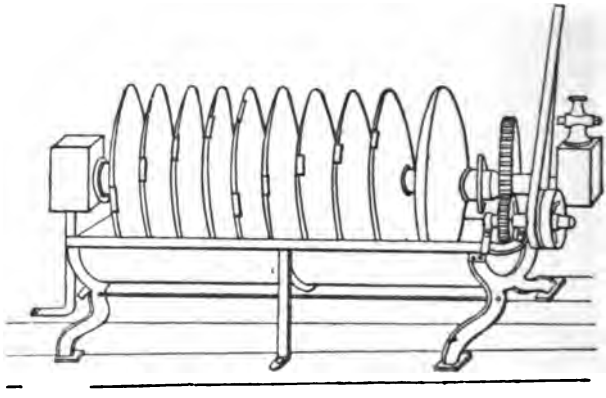


FIG. 101.—WETZEL EVAPORATOR.

then concentrated in a modification of the "Wetzel" evaporating-pan (originally introduced for sugar-boiling), constructed by Chenaillier, Paris, and others. This *évaporateur universel*, as it is termed, which is very economical and effective, is shown in Fig. 101, and consists essentially of pairs of saucers set edge to edge upon a hollow central revolving shaft, through which steam passes to the interior of the saucers (the waste steam from a high-pressure engine will do); the lower edges of the saucers dip in a jacketed trough of the liquid to be evaporated, and when they are revolved, layers of this are brought up and speedily concentrated on their surface. It may also be worked in a vacuum as shown in Fig. 102.

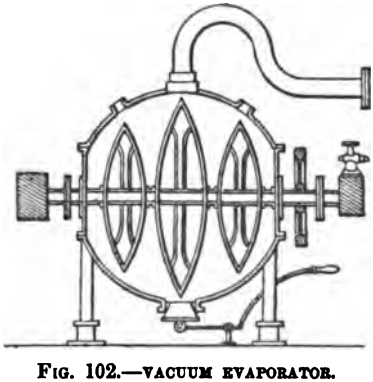


FIG. 102.—VACUUM EVAPORATOR.

Evaporation is continued to 44° Tw. (26° B., or 1·220 sp. gr.), when the glycerin is of a brownish colour, and known as "raw," in which state it is sold for many purposes. In general practice the further purification is conducted as follows. The raw glycerin, sp. gr. 1·240–1·245, is first distilled, and the distillate heated in a jacketed pan with that kind of animal charcoal known as ivory-black, and again distilled; this alternate treatment is repeated as often as may be necessary. The distillation is performed with superheated steam in a copper still provided with copper fractional condensers (the same as shown in Fig. 93, p. 355, but omitting the right half of the apparatus, including the tanks G), the still being also heated externally; the operation is performed at as low a temperature as is consistent with distillation, usually about 440° F. (227° C.). The number of distillations depends upon the quality of the raw glycerin and the purity of the product demanded. Of the 6 runs, Nos. 1, 2, and 3 usually give pure glycerin, while the dilute condense-products from Nos. 4, 5, and 6 are generally returned to the still, though occasionally concentrated in an *évaporateur universel*, or in a vacuum-pan. Some stills hold as much as 6 tons, but they are usually smaller, and in all cases the process is conducted very slowly. A form of still and condenser much used on the Continent of Europe is outlined in Fig. 103. External heat and injected superheated steam are used to effect distillation. The still A has an unusually large head B, and the gooseneck C is provided with a catch-box D, in case the still-contents should, as sometimes happens, boil over; the fractional condensers E are upright cylinders with longitudinal partitions F running

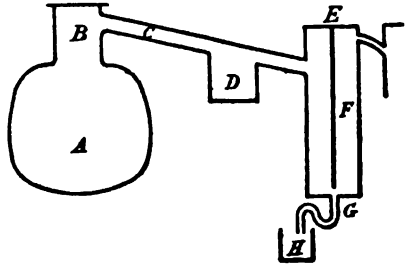


FIG. 103.—STILL AND CONDENSER.

nearly their whole length; the condensed products run out through G into receptacles H. The whole apparatus is of iron, and usually made to distil ½ ton at a time; in some cases the process is conducted continuously, with a properly arranged feed.

At the first general meeting of the Society of Chemical Industry, the President called attention to the enormous waste of glycerin in the manufacture of soap as then (and at present) conducted; he also pointed out that the growing demand for this substance for the preparation of dynamite and the various explosive derivatives of nitro-glycerin, was causing a great increase in its price. One of the earliest attempts to extract glycerin from the spent lye of the soap-house was a patent by H. Reynolds, June 10, 1858, for concentrating it, and distilling off the glycerin by superheated steam between 380° and 400° F. (193½–204½° C.); the large quantity of sodium salts, especially sodium chloride, was found, however, to be an almost insuperable difficulty. From that time the subject has been pursued with varying success by both British and Continental soapmakers, and although a large number of patents on the subject have been taken out during the last 4 years, very few of them are actually worked in practice. The history of many of them is only another instance of the well-known fact that it is far easier to obtain a patent than to make a discovery.

The "spent lye" contains water, glycerin, sodium chloride, sulphate, and carbonate, a small quantity of caustic soda, together with albuminous, resinous, and soapy matters, whose amounts vary with the nature of the materials which have been saponified. C. T. Kingzett (Jour. Soc. Chem. Ind., i. 78) gives the following as its average composition after concentration to 72° Tw. (38° B.), the gallon being composed of—

Water	7·53 lb.
Glycerin	2·04 „
Salts	2·78 „
	12·35 „

and the same authority gives the following analysis of the salts as deposited from the lye on concentration—

Sodium chloride	78·12
Sodium sulphate	8·61
Insoluble organic matter	0·22
Glycerin and organic matter	3·55
Water	7·50
Alkali (calculated as sodium carbonate) ..	2·61
	100·61

The objects of the various patented processes may be reduced to 3, viz.—

1. To remove albuminous, resinous, and soapy matters from the lye, or destroy them.
2. To facilitate the removal of the salt, either by diminishing in some way the solubility of the sodium chloride, or substituting for it another which may be more readily and profitably removed.
3. To economise the cost of concentration to the point where the product may be either used as crude glycerin, or purified by distillation.

To effect the first object, a large number of "coagulating agents" have been proposed, and in most cases abandoned. The process by which the first successful production of crude glycerin from soap lye was effected on a manufacturing scale was patented on March 31, 1879, and was for some time worked, by Thomas, Fuller, and King. It consisted in boiling the concentrated lye with a fatty acid, which removed all the caustic and carbonate of soda, and simultaneously "coagulated the albumen." Among the other "coagulating agents" proposed at various times have been—albumen itself, blood, gelatin, salts of alumina, salts of chromium, caustic lime, calcium chloride, tannic acid, &c. Under the second head may be mentioned the use of hydrochloric acid gas, to render the salt more insoluble, and of carbonic acid gas to convert the alkaline soda salts into bicarbonate; also the proposal to employ sodium or potassium sulphate, or caustic alkali, instead of sodium chloride in "salting out" the soap.

Thus, Clolus saturates the cold liquor with hydrochloric acid till it is neutralised, and the neutral clear liquid is evaporated to a density of 57° Tw. (32° B.). The glycerin present is further freed from impurities by blowing in air (with heat) to remove the moisture, whereby also the salts in solution are precipitated, being very slightly soluble in anhydrous glycerin. The glycerin is drawn off by a hydro-extractor or by dialysis.

Depoully and Droux combine the glycerin present in spent lye with acids to form insoluble glycerides, and the foreign matters are eliminated by washing with water. The glycerin is recovered by saponifying the glycerides.

Payne first saturates the unspent alkali with hydrochloric acid, then separates albuminous and gelatinous matters by a reagent such as tannic acid, next filters and evaporates the liquid containing the glycerin and sodium chloride, and finally distils the glycerin.

Versmann separates a large percentage of the salts present in the lye by boiling down and raking them out as they become insoluble; then the concentrated solution is cooled and carbonic acid gas is passed through it till all the carbonate and caustic soda are converted into bicarbonate, which is much less soluble in glycerin, and can be easily removed by filtration. The sodium chloride still present in the glycerin is eliminated by dialysis.

Ruffin causes separation by action of superheated steam in a centrifugal hydro-extractor.

Battershall employs neutralisation of the alkalies present, evaporation, distillation, and centrifugal machines. The lye is heated by steam coils in tanks, and the alkali is neutralised by adding 5 per cent. sulphuric acid (1 part water, 1 part acid at 168° Tw.); the bulk is reduced to $\frac{1}{10}$ by evaporation; lime carbonate is next added to the strained liquor, and concentration is continued till a syrupy consistence is attained; the mass is then put into a hydro-extractor to separate the glycerin from the salts. Sometimes methylic or ethylic alcohol is added to the glycerin as it leaves the centrifugal.

Allan thoroughly spends the lye, then neutralises with mineral acid; settles, adds solution of alum, or pyroligneous acid, or lime chloride; settles again, evaporates clear upper liquor to concentration, and finally distils.

Brochon treats the lye with sodium chloride to absorb excess of water, settles, and filters off the clear liquid; this is treated with slight excess of sulphuric or hydrochloric acid. The part thus rendered insoluble is precipitated by albumen, gelatin, or any chloride or soluble metallic sulphate in the cold. The clear liquor is filtered off, boiled, rendered alkaline with milk of lime which precipitates the metallic salts, settled, treated with just sufficient soda bicarbonate to precipitate the lime, and neutralised with hydrochloric acid. The liquor is boiled down in a series of pans to concentrate the glycerin and cause the salts to crystallise out. Finally the glycerin is distilled.

Lawson and Sulman first evaporate the lye down to a density of 28°–32° Tw. (18°–20° B.), and cool it. The supernatant soapy matters are then skimmed off. Next the albuminous matters are removed by heating and adding an astringent metallic salt. The removal of the albuminous matters at this stage prevents their decomposition by further evaporation, and affords a purer and more concentrated glycerin. The alkalinity of the liquor is first neutralised by acid; then the liquor is treated with a slight excess of calcium carbonate, and boiled, to precipitate chromic salts added as decolorisers, and to neutralise any remaining acid, at the same time sending down the metallic salt used to precipitate the albuminous matters. The resultant liquor is filtered and evaporated to yield the glycerin.

Bangs cools the glycerin liquor from saponification, to separate most of the fatty acid as an emulsion, and evaporates in a tin- or lead-lined vessel, with occasional addition of white stearic acid, which has the effect of decomposing the lime salts of the volatile organic acids. As lime stearate is formed, the supply of stearic acid must be renewed. The impurities go with the stearate, and the glycerin is found pure beneath

a crust of stearic acid and stearate when the evaporation has been continued till the density is 36°–40° Tw. (22°–24° B.) at 60° F. (15½° C.).

Payne boils the lye with ¾ its weight of caustic lime, to precipitate the fatty and resinous matters; neutralises with excess of hydrochloric acid, and boils; finally excess of acid is removed by lime, and the product is evaporated and distilled.

Many ingenious methods of economic concentration of the lye have been devised. It is generally considered advisable that an ordinary concentrating pan, if used, shall be fired at the side, and not from beneath, to avoid cracking it from the deposition of salts. A very effective evaporator for this purpose is that patented by Thomas and Domeier, June 4, 1881. From a rose D, Fig. 104, at the top, the liquid trickles down over the inclined shelves E, during which it meets an upward current of hot air introduced through the pipe F, whose mouth is protected by a suitable cover. The hot air and watery vapour are drawn off by an exhauster through the pipe G. The same patent covers one of the few commercially successful processes for the preliminary purification of the crude glycerin. The lye is concentrated, then treated with a slight excess of mineral acid, and allowed to stand some hours, after which the scum is removed, and the liquid is exactly neutralised with alkali. This crude glycerin is then washed by being mechanically mixed with about ⅓ of its bulk of some suitable solvent,

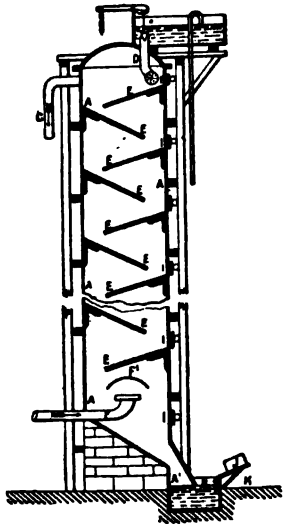


FIG. 104.—EVAPORATOR.

which will dissolve out the impurities. For this purpose the patentees use "a hydrocarbon obtained from the distillation of coal tar, petroleum or other mineral oil, carbon bisulphide, amylic alcohol, ether, or other suitable solvent in which the glycerin is practically insoluble."

Tardani uses the liquor from the first saponification, containing 4 per cent. of glycerin, 3 or 4 times, instead of fresh water; the liquor thus becomes highly charged with glycerin, and economy is attained in its extraction.

O'Farrel follows in the same lines, and when a maximum of glycerin is contained in a minimum of lye, the solution is evaporated to make as much salt as possible crystallise out, and then the glycerin is dissolved or distilled out from the remainder.

Another proposal* is to mix the concentrated crude glycerin with more than its own volume of methyl or ethyl alcohol, and separate the salts by filtration. Lead oxide, or soluble lead salts, are added to remove the last traces of chlorine, and the final filtrate is distilled, to drive off the alcohol, which also carries off some of the volatile fatty acids as ethers, thus effecting a further purification of the glycerin.

It is usual to employ a centrifugal machine or a filter-press to extract the last traces of crude glycerin from the salts deposited during concentration. Other methods than distillation also have been proposed for purifying crude glycerin, such, for example, as that of H. Flemming, of Kalk, who has patented in Germany (No. 12,209) a process for removing glycerin from spent lye by dialysis, a membrane of parchment-paper serving as the diaphragm through which the crystalline salts present in the glycerin diffuse themselves.

It has been the opinion of many who are well qualified to judge that all these processes for the recovery of glycerin from spent lye would soon be superseded by the removal of glycerin from fats prior to their saponification. Such a proceeding is by far the more scientific, but the great practical

* Eng. Pat. 2326, 1883.

objection to it is the slight inferiority, *in appearance only*, of the soap ultimately produced, and this seems to have so far deterred manufacturers from making the change. This process was worked out by Michaud Frères of Paris, and has been patented in most countries. In America it is now extensively worked. It consists essentially in resolving neutral fat into fatty acids and glycerin by the action of water and a minute quantity of zinc oxide (or, in another modification, of magnesium carbonate), under pressure, and it is thus described in the English patent (Oct. 27, 1882):—

"For this purpose the fatty matter is subjected in a close vessel to the action of steam at a pressure of 100–130 lb. per sq. in. and at corresponding temperature, in presence of $\frac{1}{4}$–$\frac{1}{3}$ part of its weight of water, and $\frac{1}{8}$–$\frac{3}{8}$ per cent. of its weight of the oxide of zinc known commercially as zinc white, or a like proportion of zinc powder or zinc grey, which is a residue in the treatment of zinc, being a mixture of zinc with its oxide.

"The close vessel and the decanting tanks are arranged in the same way as those used for saponification by the lime process, and the system of operation is the same after the steam pressure has been applied for 3 or 4 hours, according to the character of the material operated upon and the quantity of zinc or its oxide employed. The very small proportion of mineral substance used is sufficient for dispensing with the acid treatment applied for decomposing lime soap, and the product obtained, consisting almost exclusively of acid fat, can be converted by the acids usually employed into soap or candles. In soapmaking, the dissolving powers of the caustic alkalies remove all objections to the presence of the zinc, if it should be used in excess. The reducing power of the zinc powder prevents discoloration of the acid fats, such as results from the ordinary treatment, and the use of zinc as described dispenses with that of the agents usually employed for decomposing the fatty salts, so that there is great economy of labour in the process, and great diminution of tear and wear of the plant."

The following is an account of the details of an experiment made in a large American factory:—

"The experiment was made as follows:—Jan. 15, 1884, placed in digester 8096 lb. of tallow; Jan. 16, pumped from settling tanks the free fat acids, made same into soap, and boiled to a good dry curd; removed same from pan Jan. 17 and weighed carefully, and found as a result that we received 12,666 lb. of good marketable soap, or a percentage of 63·39 per cent. of fat acids against the usual yield of 65 per cent., showing an actual gain of moisture by using our process of 1·61 per cent."

The glycerin thus produced finds a ready sale as it runs from the evaporators; and from it, as "crude," 96 per cent. of pure glycerin can be obtained.

Many modifications of and substitutes for this process have been adopted or proposed, some of which deserve mention.

Baujard relies upon the action of "oxygen and hydrogen in the primitive state," brought about by steaming the fatty matters with water in a digester, and adding metallic zinc in a fine state, pressure being maintained at 125–150 lb. per sq. in. The water is partially decomposed.

Field mixes the fatty matters with water in a cylindrical, horizontal vessel, surrounded by a heating space, and fitted with agitators. The contents after treatment are discharged in fine streams, to facilitate dissipation of the steam. On settling, the glycerin and water go to the bottom.

Burghardt places the raw fat in a jacketed retort, heated by steam at 260°–360° F. (127°–182° C.). When the fat begins to volatilise, cold air is blown upon the vapours, and drives them into a condensing apparatus, where they are collected over water; or direct into caustic lye for soap-making.

Numerous other patentees avail themselves of superheated steam, and differ only in the form of apparatus used.

Rumble and Sear mix the fats with 5–8 per cent. of a zinc or magnesium salt of a fatty acid, and about $\frac{1}{3}$ of water,

and subject the whole in an autoclave to steam at 120–130 lb. per sq. in., the temperature being maintained at 368° F. (187° C.). The glycerin is thus set free, and can be recovered in the usual way. The soaps formed are decomposed by a mineral acid, and the metallic salts recovered by an alkaline solution.

Marix decomposes the fat by mechanical means in the shape of a finely divided neutral substance (as pipeclay) acting by abrasion on the cellular membrane, liberating the glyceral oxide so that it may combine with the water present, and freeing the fatty acids. This is effected *in vacuo*, and the various constituents are drawn over and separately condensed.

Although evaporation and distillation are the usual methods of purifying glycerin, the action of cold upon more or less dilute glycerin is sometimes employed in conjunction with them, especially by Sarg, at Vienna. When an aqueous solution of glycerin partially freezes, the frozen mass contains more water than the remaining liquid, hence some amount of concentration may be thus effected. The following table gives the freezing-points of such mixtures:—

Glycerin per cent.	Sp. Gr.	Freezes.	Glycerin per cent.	Sp. Gr.	Freezes.
10	1·024	− 1° C.	60	1·159	Below − 35° C.
20	1·051	− 2°·5	70	1·179	
30	1·075	− 6°	80	1·220	
40	1·105	−17°·5	90	1·232	
50	1·127	−31°·3	94	1·241	

Another authority gives:—

Glycerin solution sp. gr.	10° B.	12° B.	14° B.	15° B.
Melting-point ..	− 9° C.	− 13° C.	− 18° C.	− 21° C.

In January 1867, some glycerin sent in tin cans from Germany to England froze into pea-sized octahedral crystals; these, while melting, had a constant temperature of 45° F. (7·2° C.), but would not freeze again even when cooled to 0° F. (− 18° C.). According to Werner, commercial glycerin

will freeze more readily if chlorine gas be passed into it. In purifying glycerin by cold, the whole mass is cooled to nearly 32° F. (0° C.), and some crystals of solid glycerin are added; almost the whole mass solidifies on agitation, and a centrifugal machine is used to separate the solid from the liquid parts. Treated in this way, glycerin at 48° Tw. (28° B., or 1·240 sp. gr.) yields crystals which, when melted, are 54° Tw. (30½° B., or 1·270 sp. gr.).

Pure glycerin is a viscid, colourless, and transparent liquid, with an intensely sweet taste, soluble in water in all proportions, in alcohol, chloroform, and carbon bisulphide, but not in ether; its sp. gr. is 1·267; it solidifies at – 32° F. (– 35·5° C.) to an amorphous mass. When distilled, it is said by G. Th. Gerlach * to boil so quietly and uniformly at 554° F. (290° C.) that it may be used for determining that point on high-temperature thermometers. According to Bolas, however, at 12·5 *mm.* pressure it boils at 355° F. (179½° C.), and at 50 *mm.*, at 410° F. (210° C.), while Heminger gives 354° F. (179° C.) as its boiling-point under 20 *mm.* It burns with a clear flame like oil, if there be free access of air and a high temperature for kindling it.

The chemical constitution of glycerin, and its relationship to fatty bodies, were fully explained in Chap. II. It remains to notice some of its chief physical properties, the chemical tests for it, some of its uses, and its possible adulterants.

Next to water, glycerin is the most powerful solvent known. It dissolves bromine, iodine, and carbolic acid better than water does. Klever gives a long table of the solubilities of different substances in 100 parts of glycerin, from which the following are taken:—93 sodium carbonate, 40 alum, 25 green vitriol (ferrous sulphate), 20 lead acetate, 20 sodium chlorate, 0·50 quinine and other alkaloids, 1·9 iodine, 0·20 phosphorus, 0·10 sulphur.

With baryta, strontia, and lime, it forms compounds insoluble in water, not precipitable by carbonic acid. Anhy-

* Dingl. Polyt. Journ., 255, 208, 1885; abstracted in Journ. Soc. Chem. Ind., iv. 226.

drous glycerin dissolves caustic potash and soda, lead oxide, all deliquescent salts, potassium, sodium, and copper sulphates and chlorides, and the vegetable acids and alkaloids. It mixes with water in all proportions; the following tables will be found very useful commercially:—

TABLE OF QUANTITY BY WEIGHT OF WATER IN 100 PARTS BY WEIGHT OF DILUTE GLYCERIN AT 63½° F. (17½° C.).—[F. HOFFMANN.]

Sp. Gr.	Per Cent. Water.	Sp. Gr.	Per Cent. Water.	Sp. Gr.	Per cent. Water.
1·267	0	1·212	17	1·161	34
1·264	1	1·209	18	1·159	35
1·260	2	1·206	19	1·156	36
1·257	3	1·203	20	1·153	37
1·254	4	1·200	21	1·150	38
1·250	5	1·197	22	1·147	39
1·247	6	1·194	23	1·145	40
1·244	7	1·191	24	1·142	41
1·240	8	1·188	25	1·139	42
1·237	9	1·185	26	1·136	43
1·234	10	1·182	27	1·134	44
1·231	11	1·179	28	1·131	45
1·228	12	1·176	29	1·128	46
1·224	13	1·173	30	1·126	47
1·221	14	1·170	31	1·123	48
1·218	15	1·167	32	1·120	49
1·215	16	1·164	33	1·118	50

G. Th. Gerlach's determinations, 1885 (*loc. cit.*):—

Percentage of Pure Glycerin.	Specific Gravity.		Percentage of Pure Glycerin.	Specific Gravity.	
	At 15° C.	At 20° C.		At 15° C.	At 20° C.
100	1·2653	1·262	40	1·1020	1·101
90	1·2400	1·236	30	1·0750	1·074
80	1·2130	1·209	20	1·0490	1·048
70	1·1850	1·182	10	1·0245	1·0235
60	1·1570	1·155	0	1·0000	1·0000
50	1·1290	1·128			

According to F. Strohmer ('Chemisches Centralblatt,' 15, 397), the amount of glycerin present in its aqueous solution

may be determined by its index of refraction. The formula given, deduced by the method of least squares, is—

$$n_{(D)} = 0{\cdot}75875 + \frac{(K + 56{\cdot}569)\,D}{D\,(100 - C) + C},$$

where D = sp. gr. of anhydrous glycerin, which is 1·262 at 63° F. (17½° C.), and C = percentage of glycerin by weight. (Jour. Soc. Chem. Ind., iii. 490.)

Commercial glycerin is liable to contain various impurities, arising from its mode of preparation; also certain adulterants, of which cane-sugar and glucose are the chief. Ferdinand Jean (Jour. Soc. Chem. Ind., i. 411) has described minutely the adulterations and imperfections in the manufacture of glycerin. Glucose may be detected by the brown colour formed when the suspected glycerin is boiled with caustic soda; or by the blue colour produced when 3 drops of glycerin are boiled in a test-tube with 100–120 drops of water, 1 drop of nitric acid, and 1 *grm.* ammonium molybdate. Cane-sugar is shown by its deposition when the glycerin is agitated with chloroform, or, more certainly, by a polarising saccharimeter, since glycerin has no rotary action on the plane of polarisation. Lead is detected by sulphuretted hydrogen; lime, by the addition of alcohol and sulphuric acid, a white precipitate of calcium sulphate being formed; butyric and formic acids, by the characteristic smell of their ethers, produced by boiling the suspected glycerin with alcohol and strong sulphuric acid; oxalic acid by the addition of calcium chloride and ammonia; sodium chloride, by the addition of silver nitrate, which should give no precipitate with pure glycerin after 24 hours' standing. A rough and ready test for impurities generally is to agitate the glycerin with an equal bulk of chloroform, when they collect in the intermediate layer. Pure glycerin should not blacken when mixed, *in the cold*, with strong sulphuric acid.

Traces of glycerin present in other substances may be detected by the formation of formic ether (which smells of

peach-blossom), produced by boiling glycerin with manganese binoxide, alcohol, and sulphuric acid.

The uses of glycerin are very numerous, and are almost daily increasing in number. Its applications in pharmacy are nearly endless; it is used wherever a substance requires to be kept more or less moist, e. g. modelling clay, tobacco, paper for printing, adhesive gum, &c.; also in spinning, "dressing," weaving, ropemaking, and tanning. It is employed in gas-meters, and in floating compasses, to lower the freezing-point of the water therein used; it is an excellent preservative medium for meat, and for natural history specimens, to which latter purpose it was first applied in 1856 by Dr. Carpenter, F.R.S. Owing, however, to its solvent action upon calcium carbonate, delicate specimens, especially for the microscope, should be mounted in glycerin which has been standing over crushed marble. Glycerin is also of very great importance as the starting-point of other chemical products of great value, one of the most valuable of which is nitro-glycerin. For this purpose, it must contain no sodium chloride. The engineers of the Panama Canal estimated their requirements of nitro-glycerin at a minimum of 8000 tons, equal to about half that quantity of raw glycerin. Besides nitro-glycerin, two other important products are obtainable from glycerin, viz. isopropyl iodide, and allyl iodide, each of which serves as the starting-point of a large series of chemical products, many of them of great utility in the arts. They are formed by heating glycerin with hydriodic acid, thus:—

$$C_3H_5(OH)_3 + 5\,HI = 2\,I_2 + 3\,H_2O + C_3H_7I \text{ (isopropyl iodide).}$$
$$C_3H_5(OH)_3 + 3\,HI = I_2 + 3\,H_2O + C_3H_5I \text{ (allyl iodide).}$$

When oxalic acid is heated in contact with glycerin, the former breaks up into formic acid and carbon binoxide. This process is much used in the preparation of formic acid, the glycerin employed not being consumed, but merely successively decomposed and recomposed.

The total production of glycerin yearly was estimated by

Riche, in a report on the Paris Exhibition of 1878, at 10,000,000 *kilo.*, with a value of 5–6 million *fr.* The production was thus distributed:—France, 4000 tons; Germany and Austria, 1500; Holland, 900; Russia, 900; Belgium, 800; Italy, 400; England, 300; Spain, 100. In 1891 the total output was computed at 40,000 tons, 14,000 tons in soapmaking, and 26,000 tons in stearin making. Of the former, 5500 tons were made in the United Kingdom, 3500 in France, 3000 in the United States, and 2000 in Germany. Of the latter, 6000 tons in France, 3000 each in Germany and the United States, 2000 each in Holland, Austria, and Russia, 1800 each in Belgium and Italy, 1500 in Spain, 1200 in the United Kingdom, 350 in Sweden and Norway, 300 each in Switzerland, the Argentine, and Australia, 200 in Portugal, 150 in Roumania, and 100 in Greece.

CHAPTER XVII.

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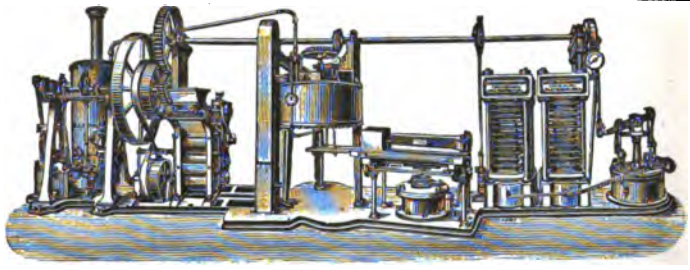

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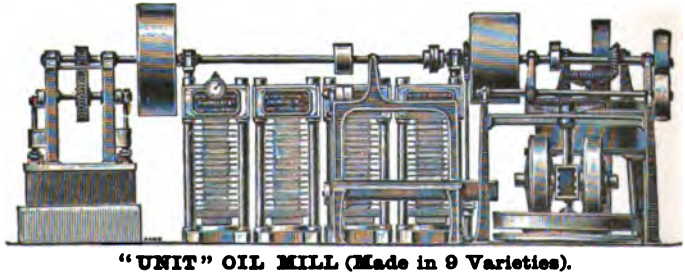

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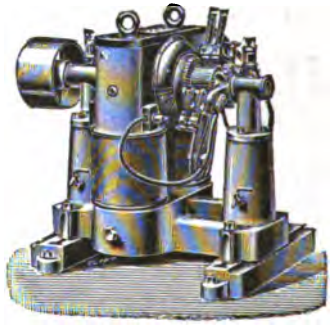

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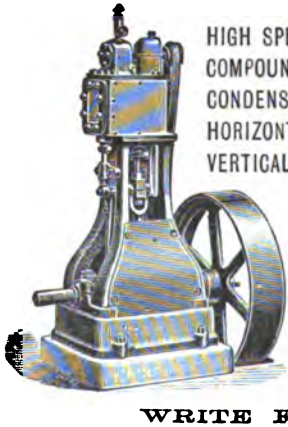

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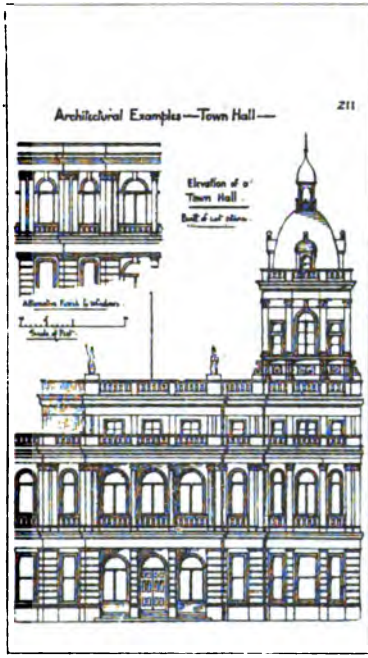

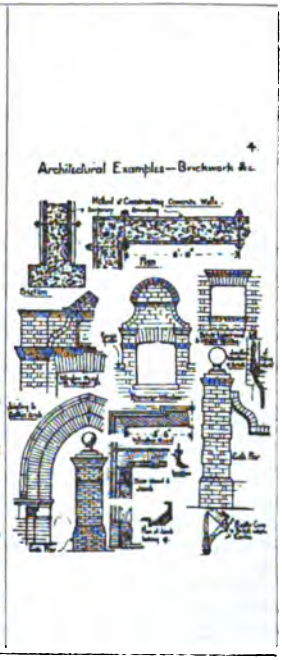

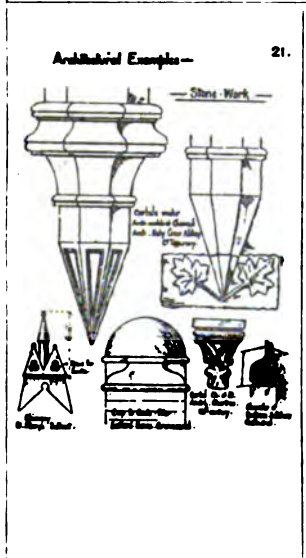

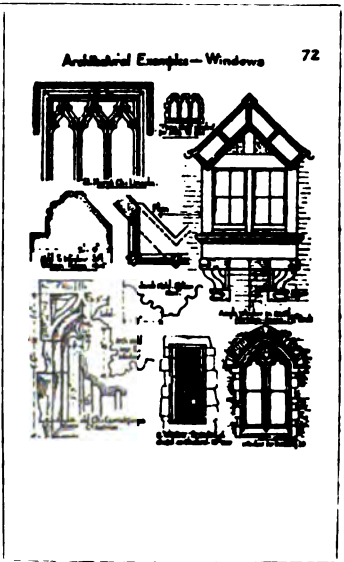

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